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MICROGRAVITY SILICON ZONING INVESTIGATION

Annual Report (July 15, 1982 through July 15, 1983)

July 15, 1983

Prepared for

GEORGE C. MARSHALL SPACE FLIGHT CENTER
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Annual Report

16. ABSTRACT

This year's effort has further defined the ground based reference for the growth of silicon by float zoning which begain under Contract NAS8-34542. A resistance heated zoner, suitable for early zoning experiments with silicon, has been designed and put into operation. The initial power usage and size has been designed for and shown to be compatible with payload carriers contemplated for the Shuttle. This equipment will be used in the definition and development of flight experiments and apparatus for float zoning silicon and other materials in microgravity.

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INTRODUCTION AND SUMMARY

This is the final report of the first year's effort on Contract NAS8-34920 "Microgravity Silicon Zoning Investigation". The contract period was 16 July, 1982 to 15 July, 1983. This contract was performed for NASA's George C. Marshall Space Flight Center and was monitored by Mr. I.C. Yates.

This contract period has further defined the ground based reference for the growth of silicon by float zoning, which was commenced on Contract NAS8-34542. A resistance heated zoner, suitable for early zoning experiments with silicon, has been designed, fabricated and put into operation. Initial power usage and size has been designed for and shown to be compatible with the MEA and Hitchhiker carriers for the Shuttle. The effort was divided into three tasks, which will be summarized below.

Task 1 - "Characterize the Silicon Molten Zone", further characterized the zoning of silicon at g=1 and 2.5 cm diameter. The growth was performed in NASA's (r.f.) Breadboard Zoner, which was modified to provide better instantaneous speed control through replacement of the translation motor controls and motors and modification of the rotational motor controls and mechanical parts of the transports. A laboratory peltier pulser was assembled to mark the growth interface of the silicon crystal, and this led to the design and construction of a prototype pulser unit. Thermal profile measuring techniques were developed using thermocouples and an I.R. microscope and the axial temperature profile of the solid-melt silicon system was profiled. Thermal oscillations in the melt were also observed directly. Analyses

were carried out on silicon slices which were partially melted and regrown at MSFC by an electron beam melting method.

Task 2 - "Comparison of Zone Heating Methods" analyzed the various ways of heating silicon rods for float zone growth for early flight experiments. This task was terminated early, when the resistance heated method showed promise for early flight hardware.

Task 3 - "Characterize the Float Zone in Small Diameter Silicon Rods" was the first ground based research for an early flight experiment for the float zone growth of silicon. A resistance heated furnace was designed, fabricated, debugged and started up. A Pt-10% Rh wire heated hot-wall furnace was able to melt 5mm diameter silicon with a power of 170 watts (at 15 v.) in 1 atmosphere of Argon and less than 90 watts (at 11 v.) in 10^{-1} torr vacuum. These are both well within the voltage and power available on the Hitchhiker. The Thin Rod Zoner was designed as a form and functional prototype of a flight furnace.

It is recommended that the ground based research be continued for one additional year, at the end of which the flight experiments and hardware will be specified. The major efforts should be:

a) Study and learn to control Marangoni flow in a silicon slice zoning experiment, requiring the fabrication of a small radiant heated apparatus.

b) Optimize the Thin Rod Zoner for control of zoning conditions. This will include optimizing the heater design and construction, optimizing the silicon chucks, adding a microprocessor control and using a zoning parameter program started in the first year.

c) Fully characterize zoning silicon at 5mm diameter and

analyze the crystal grown, as a function of zoning parameters. Use peltier pulsing to mark the growth interface. Fully characterize power and cooling requirements.

d) Further develop the Thin Rod Zoner hardware needed to specify a flight zoner at the end of the year. This will include environmental and safety needs and environmentally testing to determine any changes needed in hardware design and to avoid damage to silicon and heater parts. Vacuum and gas controls will be added. A sample changer for zoning more than one rod, as required in a microgravity research program, should be designed and the key features tested.

I. TASK 1: CHARACTERIZE THE SILICON MOLTEN ZONE

INTRODUCTION AND SUMMARY

The objective of this task is to characterize the silicon molten zone at g=1 and a diameter of one inch (2.5 cm), using state-of-the-art float zoning techniques for float zone crystal growth of silicon. This will establish the ground-based reference (at g=1) characteristics of the crystal growing system and the properties of the grown crystals and will be used to predict what will happen in microgravity.

The major areas of effort in Task 1 and the major accomplishments are summarized below, and are in the order in which they are discussed in detail in Sections I-A through I-F:

A. NASA R.F. Zoner

Based on the results of the previous contract effort (Contract NAS8-34542), modifications to the NASA Breadboard Zoner were required to assure constant and precise zoning speeds. The transverse translation motor drive train and motor control system was replaced with a state-of-the-art system and further modifications made in mechanical parts of the zoner.

B. Growth Interface Demarcation by Peltier Pulsing

Elimination of meltback striations in a resistance heated (Thin Rod) zcner (Task 3) and expected elimination of thermal oscillation striations at either g=1 or g=0 led to a need to characterize the growth interface shape and growth rate. This has successfully been done on other systems with Peltier pulsing. A laboratory system was assembled and pulsing

experiments done. A brassboard (prototype) pulser was designed and fabricated, which approximates a system which could be customized for flight experiments.

C. Measuring Melt and Solid Temperatures

Axial gradients in the solid-melt-solid float zone configuration need to be known to calculate heat flows and to correlate to analytical models. Temperatures were measured by thermocouples and by a (NASA) Barnes infra-red microscope, which was rebuilt and used on this contract.

D. Thermal Profiles in the Silicon Solid/Melt System

Using the measurements described in Section C, the axial thermal profile is presented.

E. Transients in the Silicon Melt

Thermal oscillation transients give rise to minor striations, as shown in the results of the previous contract. These are further characterized by striation etching and by the infra-red microscope.

F. Slice Zoning Experiments

The role of Marangoni (surface tension driven) flows in the silicon melt, and their effects in microgravity, are not characterized for silicon. An initial effort to characterize these was made by melting part of a thin silicon slice by an electron beam system at Marshall Space Flight Center. The initial results of this study are presented and further study recommended.

A. NASA R.F. ZONER

The NASA (R.F.) Breadboard Zoner is used to characterize

the ground-based growth of silicon. In order to predict what changes will take place by zoning in microgravity, the dynamics of the zone must be well understood. Results of the previous contract showed a possible 12 to 22% speed variation within time periods as short as 4 seconds. (Ref. 1, p. 23).

The zoner is also used to thermally profile the silicon solid-melt-solid system. Its open area around the growing crystal and cold wall allows profiling by an infra-red microscope. Measurements involving thermocouples temperature measurements and Peltier pulsing are easy in this sytem. The zoner is also used to grow crystals for the silicon slice zone experiments and thin rods for zoning studies in the Thin Rod Zoner. The initial growth program (of zoning speeds) for the Thin Rod Zoner was also done in this zoner.

The very accurate translation velocity control was assured by installing a digital translation motor control system. This system utilizes a digitally synthesized, crystal controlled clock frequency as a reference that is compared to a feedback frequency generated by an optical digital tachometer (encoder) attached to the motor. A complex feedback scheme employs 4 separate loops:

1. Digital frequency difference integrator
2. Analog rate of change
3. Digital phase comparison with analog integration
4. Transconductance power current control.

These techniques result in zero velocity error.

A "Repass" mode is provided that allows simultaneous adjustment of both upper and lower units with one control. Passing

the silicon rod, still attached to the upper chuck and the seed to the lower chuck, through the r.f. coil without opening the chamber doors and detaching the rod, is possible in this mode. This operation is necessary in the purification of high purity (high resistivity) silicon and is done on all silicon crystals and thin rods grown for the NASA effort. In the "Repass" mode, the upper rod and lower shafts translate at exactly the same speed so that the silicon rod is neither crushed nor pulled out of one of the chucks.

A "set-up" mode allows rapid translation for service use such as cleaning, changing material, etc. Additional logic provides protection for limit movements. Figure 1 shows the control panel for the motor controls on the right side of the zoner. Figure 2 shows the digital control components and the power amplifiers that were built to precisely drive the motors. Figure 3 shows the motor and gear reducer.

The rotation motor speed controllers were also replaced, with Electrocraft model E-652 amplifier/controllers. New panel controls were also installed (see Figure 1). Rotation drive belts were also replaced.

The mechanical drives for translation were upgraded and modified. In both the upper and lower transports, the Saginaw ball-bearing lead screw drive had the lead screw bearings (at the ends of the lead screw) and ball bushings replaced. A provision was made that will allow a gear box to be added in series to the present gear box for very slow experimental zoning speeds. The seals of the upper and lower shafts (where they go into the zoner chamber) were cleaned and worn parts replaced.

In order to accomodate peltier pulsing, a current must be passed through the crystal, thereby requiring that one end of

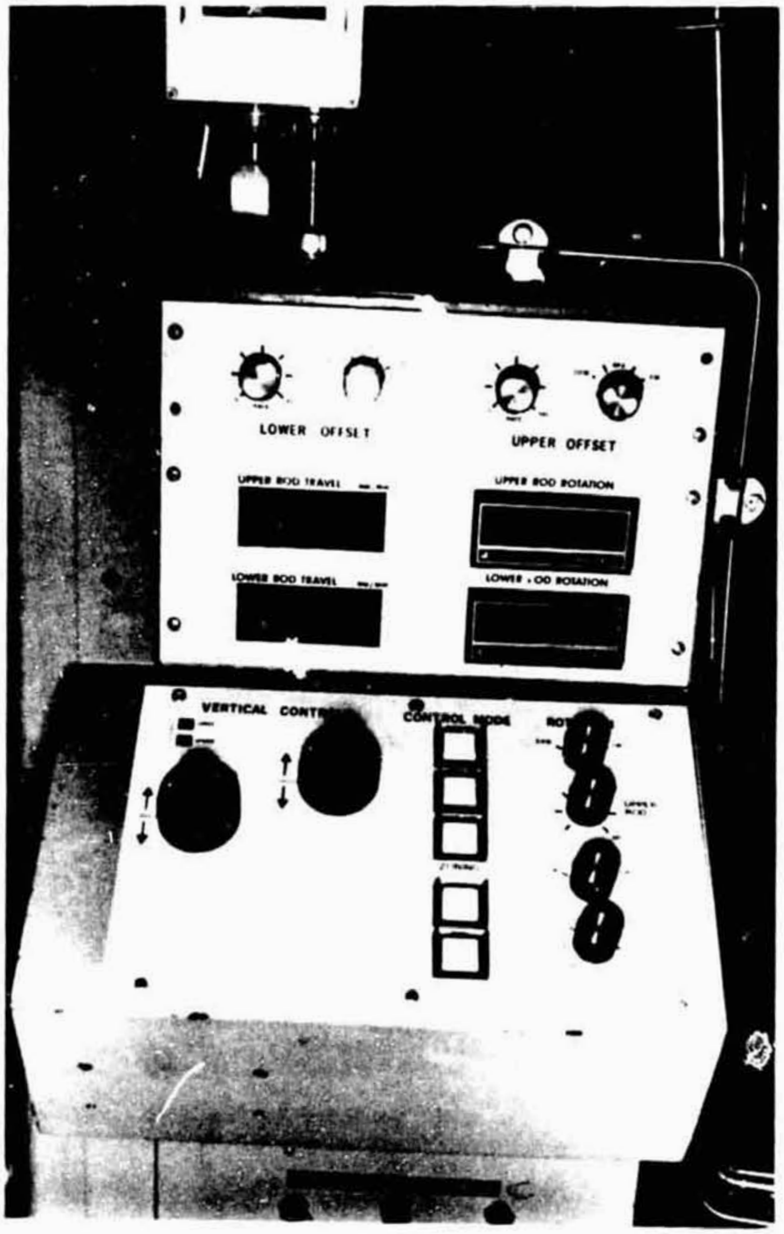

Figure 1. NASA Breadboard Zoner Motor Controls

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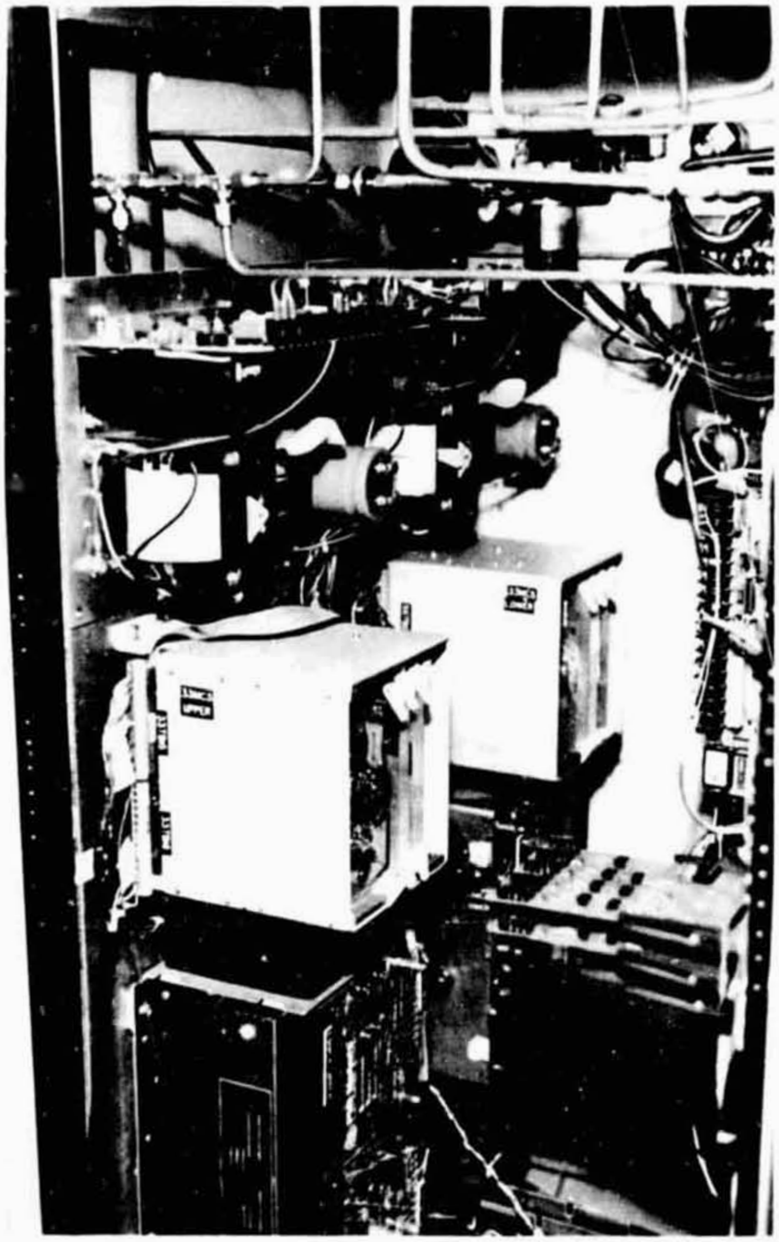

Figure 2. Motor Control Circuitry
Located in control cabinet beside the r.f. zoner

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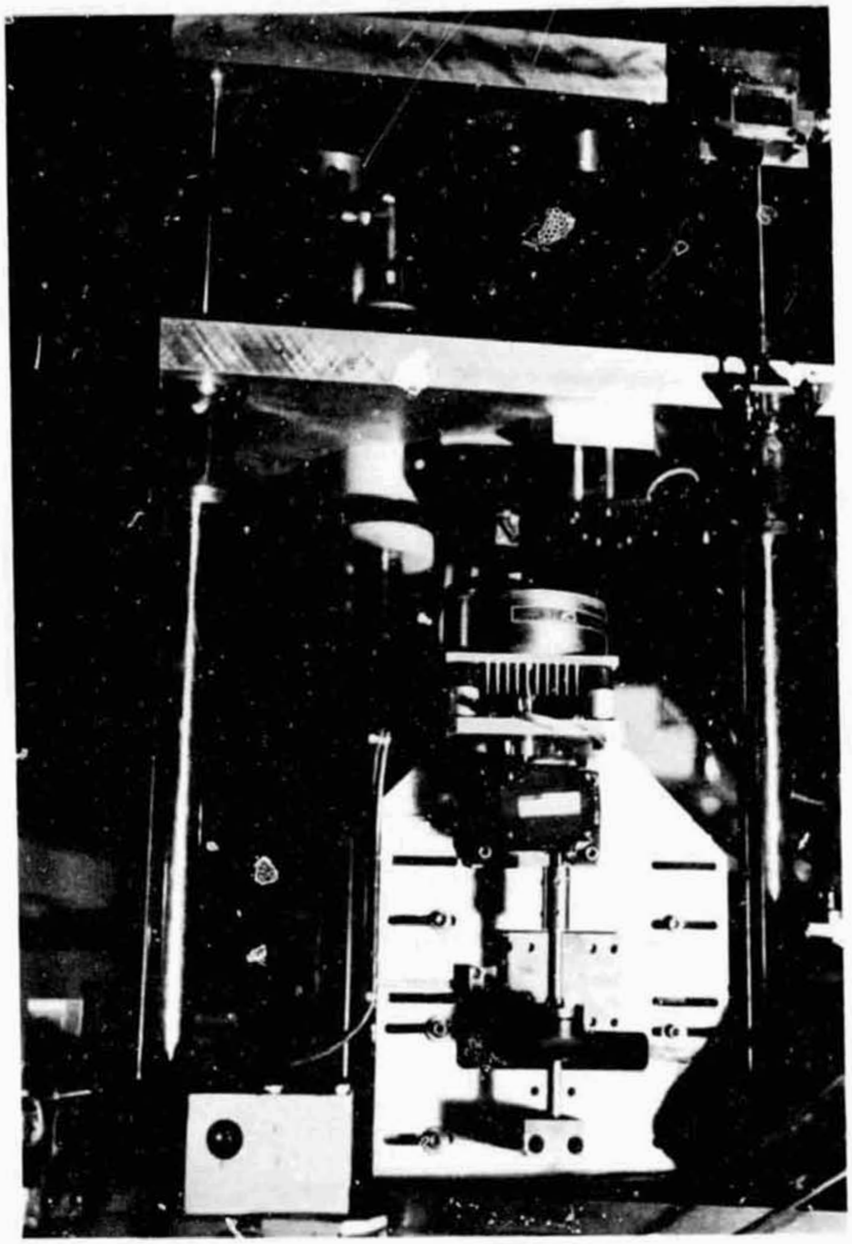


Figure 3. Upper Translation Motor and Drive Train

The motor is a printed armature low inertia motor, made by PMI, model U12M4 with 1/3 hp. It has a 5000 line optical encoder built in. The gear reducer is a 25:1 ratio by Boston Gear (model WA309-A25K).

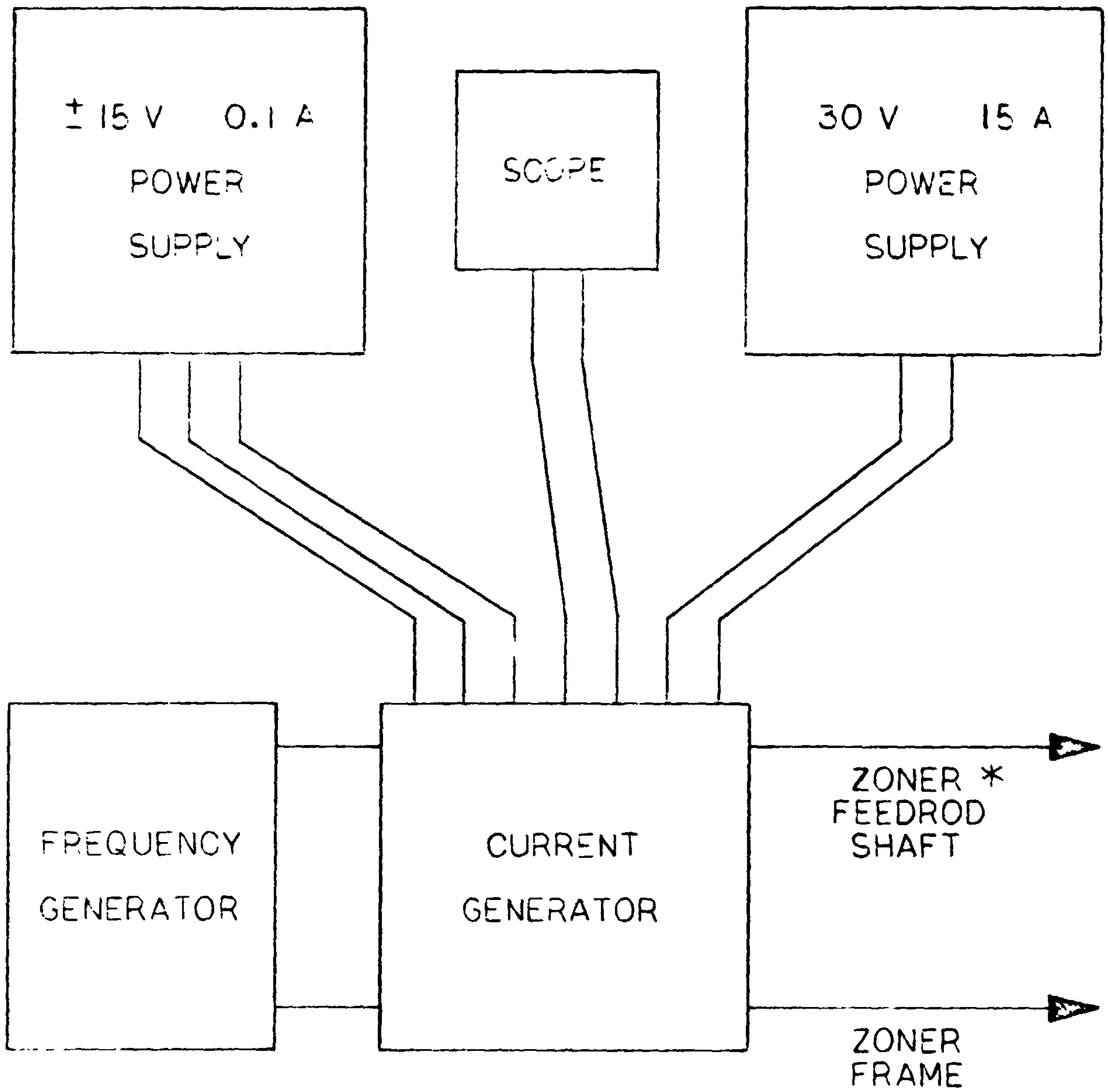
the crystal be not grounded (both are usually grounded in a zoner). The feedshaft (upper translation shaft) was electrically isolated for this need.

B. GROWTH INTERFACE DEMARCATION BY PELTIER PULSING

Decoration of the growth interface shape can be done by Peltier pulsing the melt-growth interface. This rapid melting, followed by a rapid refreezing, preferentially segregates out impurities, which will be preferentially etched by a striation etch (see Ref. 1, p. 17 for etching procedure). This will be necessary when meltback and thermal oscillation striations are eliminated. After the bench unit was assembled and successfully used, a unit specifically built as a dedicated pulser was designed and constructed, which is equivalent to the laboratory model of a flight prototype.

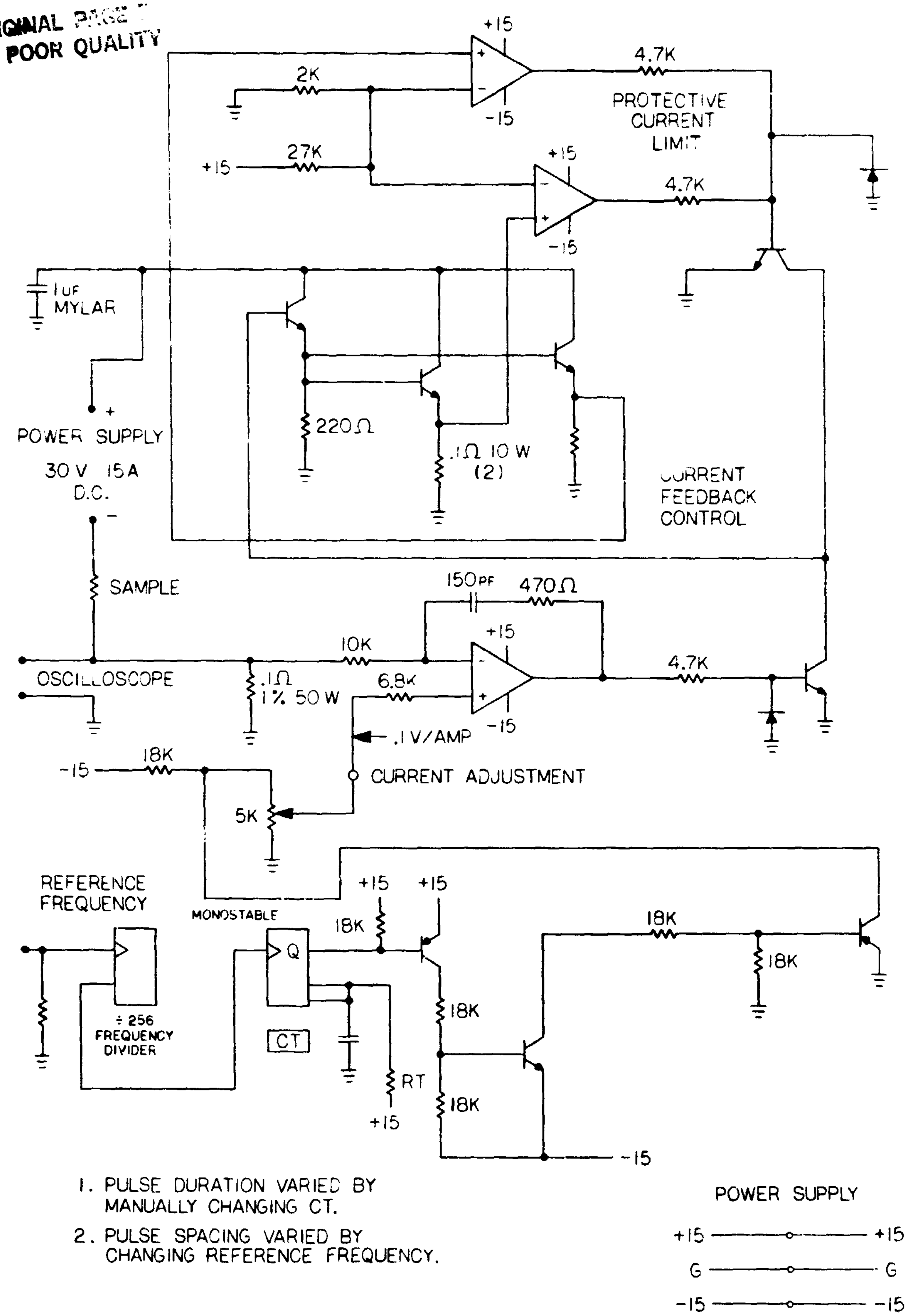
The bench unit was assembled with laboratory "black box" units and circuitry for changing pulse durations and spacings. It is similar to the type of units used by D. Larsen (Grumman) for his magnetic metal materials work. The bench unit is composed of a pulsed constant current generator with peripheral bench test equipment utilized to provide power and a frequency reference. An oscilloscope is used to provide a precise means of measuring the pulse current amplitude and pulse duration. A frequency counter was used to establish the appropriate frequency required to obtain the desired pulse spacing. Figure 4 is a block diagram of the test arrangement; Figure 5 is a schematic diagram of the current generator.

In order to pass sufficient current through the crystal



* THE ZONER FEEDROD SHAFT IS ELECTRICALLY ISOLATED FROM THE ZONER FRAME.

Figure 4.



1. PULSE DURATION VARIED BY MANUALLY CHANGING CT.
2. PULSE SPACING VARIED BY CHANGING REFERENCE FREQUENCY.

Figure 5.

CIRCUIT DIAGRAM OF PELTIER EFFECT PULSER AS USED ON R.F. ZONER

to allow for a good pulsed melting, the crystal must be low resistivity. This is done by doping the ingot to be zoned and pulsed with Gallium in the range of 10^{17} atoms/cm^3. This is accomplished by doping at the seed with the right amount of elemental gallium and then zoning. Since Ga is preferentially zoned out of the seed region, the resistivity in the seed region becomes too high. It was necessary to dope the seed portion with 10^{17} atoms/cm^3 of boron, which is not preferentially removed, since its effective segregation coefficient is about 0.95.

A program of pulse durations, spacings and currents was scheduled and crystals grown with this sequence. Table 1 gives the program sequences. The crystals are then slabbed lengthwise, polished and striation etched (the procedure is given in Ref. 1). The periodicity of long times (spacing) between pulses can then be seen with the naked eye. An example of the etching pattern is shown in Figure 6.

If peltier pulsing is to be used in flight experiments, it must use the available shuttle power, accomodate the requirements of the Hitchhiker Shuttle carrier and be done within a reasonable size and weight limitation. This requires operating on 24 v.d.c. and not translating current spikes back into the shuttle power lines that would effect other equipment. An instrument which has the flexibility needed for experimentation and which is a prototype of a unit to be designed for flight was preferred over buying larger laboratory supplies, coupled with the appropriate circuitry, such as the bench unit described above. A programmable pulsed current source (peltier pulser) was designed, using basic electronic components.

Table 1 Peltier Pulsing Experiments

		Pulse Width	Spacing	# Pulses	Pause	Repeated	Comments
	amps	ms	sec		sec	n-times	
1st Exp.	3	10	20	7	0	0	
	3	100	20	7	0	0	
	3	1000	20	7	0	0	
	7	10	20	7	0	0	
	7	100	20	7	0	0	
	7	1000	20	7	0	0	
	15	10	20	7	0	0	
	15	100	20	7	0	0	
	15	1000	20	7	0	0	
	20	100	20	7	0	0	
	20	1000	20	7	0	0	
	1	1000	20	7	0	0	all sequences done
2nd Exp	15	1000	2	5	10	15	Leading edge of
Plan	15	330	2	5	10	15	pulse in decorated
	15	100	2	5	10	15	crystal 100 msec.
	15	10	1	10	10	15	Therefore, pulses
	7	1000	2	5	10	15	should be
	3	1000	2	5	10	15	$\geq$ 100 msec.
3rd Exp.	15	1000	20	3	60	2	3 amps-pulses
Plan	15	3000	20	3	60	2	heat seed to
							glow *.
	15	330	20	3	60	2	2 amps-seed
	15	100	20	3	60	2	did not glow*
	7	1000	20	3	60	2	
	3	1000	20	3	60	2	

* Could not pass more current due to too high resistivity in the seed.

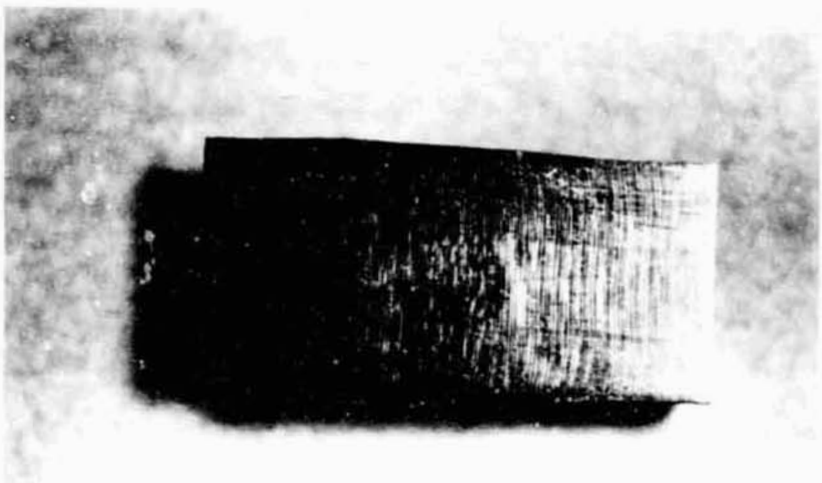


Figure 6. Peltier Pulsed Crystal

Lines show periodicity of 1 to 15 amp pulses.
Lines are brought out by striation etching.

The programmable rulsed current source (peltier pulser) provides an accurate, high impedance, constant current source that can be operated at d.c. or can be programmed to supply pulses of any desired duration and amplitude at any desired spacing. High impedance is guaranteed due to the use of feedback control and high gain.

The current generator is programmed via a user-friendly-menu format. The microprocessor will query the user for the data required. Once the data is entered, pressing the "start" key will cause the microprocessor to enter a hold state with appropriate message; pressing "start" once again will cause the program to execute. The program execution can be interrupted at any time by pressing the "pause" key. The program can be continued by pressing "start". A new Data Entry cycle is initiated by pressing the "reset" key. During program execution, the "current on" indicator will light whenever a current pulse is generated. This indicator will be lit continuously in D.C. (direct current) mode. The microprocessor will monitor the load for a compliance voltage error; this condition occurs when the load impedance is large enough to cause the load voltage to exceed the available power supply voltage. Under this condition an error message is displayed, the pulse is terminated, and the program is halted.

Figure 7 shows the block diagram schematic of the programmable pulsed current source (peltier pulser), while Table 2 gives the specifications. The constructed unit is shown in Figures 8 (front panel view) and 9 (electronic components in a rear view).

The concept of a programmable pulser is readily adapted

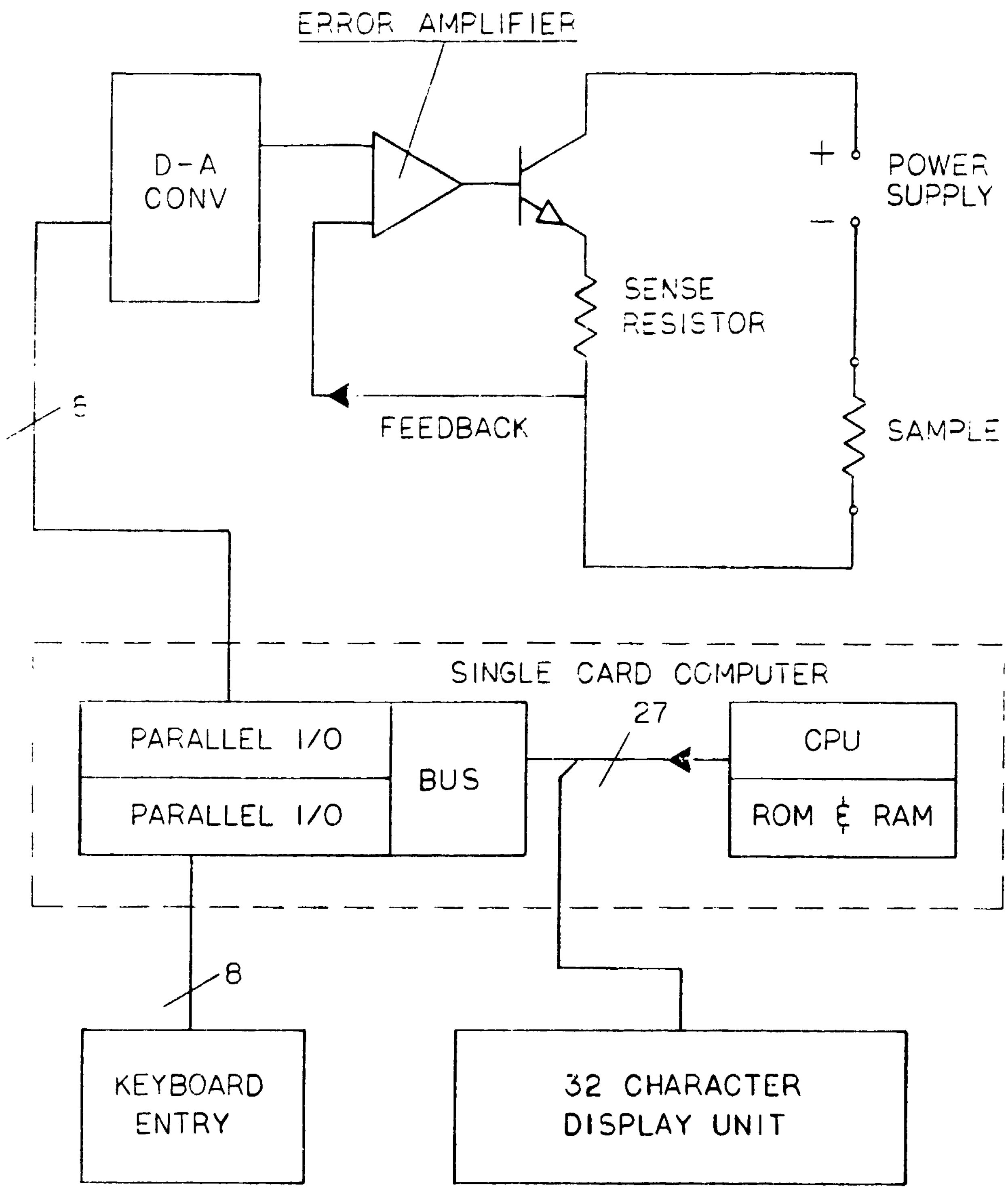


Figure 7.

BLOCK DIAGRAM BENCH MODEL

PROGRAMMABLE PULSED CURRENT SOURCE

Table 2 Preliminary Specification & Operating Description

Programmable Constant Current Source

1.	Current capability, increments of 0.1 Amp [1]		15	Amps
2.	Compliance voltage	(15 Amps) [1]	20	Volts
		(.1 Amp)	28	Volts
3.	Output impedance	(15 Amps)	>1	Megohm
4.	Minimum "ON" time of current	(15 Amps)	1	mS
5.	Rise time (10% to 90%), and current [2]		<50	uS
6.	Overshoot	Amplitude, any current	<5%	
		Time, any current	<50	uS
7.	Undershoot	same as (6)		
8.	Maximum duty cycle		D.	C.
9.	Display	32 character LCD Alphanumeric 2 lines of 16 characters		

10. Control Microprocessor

    SBE Single Board Computer, 6809 MPU.

ROM	4K
RAM	2K
Parallel	I/O 40 lines

11. Keyboard -- 10 numeric plus 6 functions
12. Compliance voltage monitor -- notifies the user when a compliance overload occurs.

[1] Compliance voltage & max current are options selected by user.

[2] Programmable rise time is optional.

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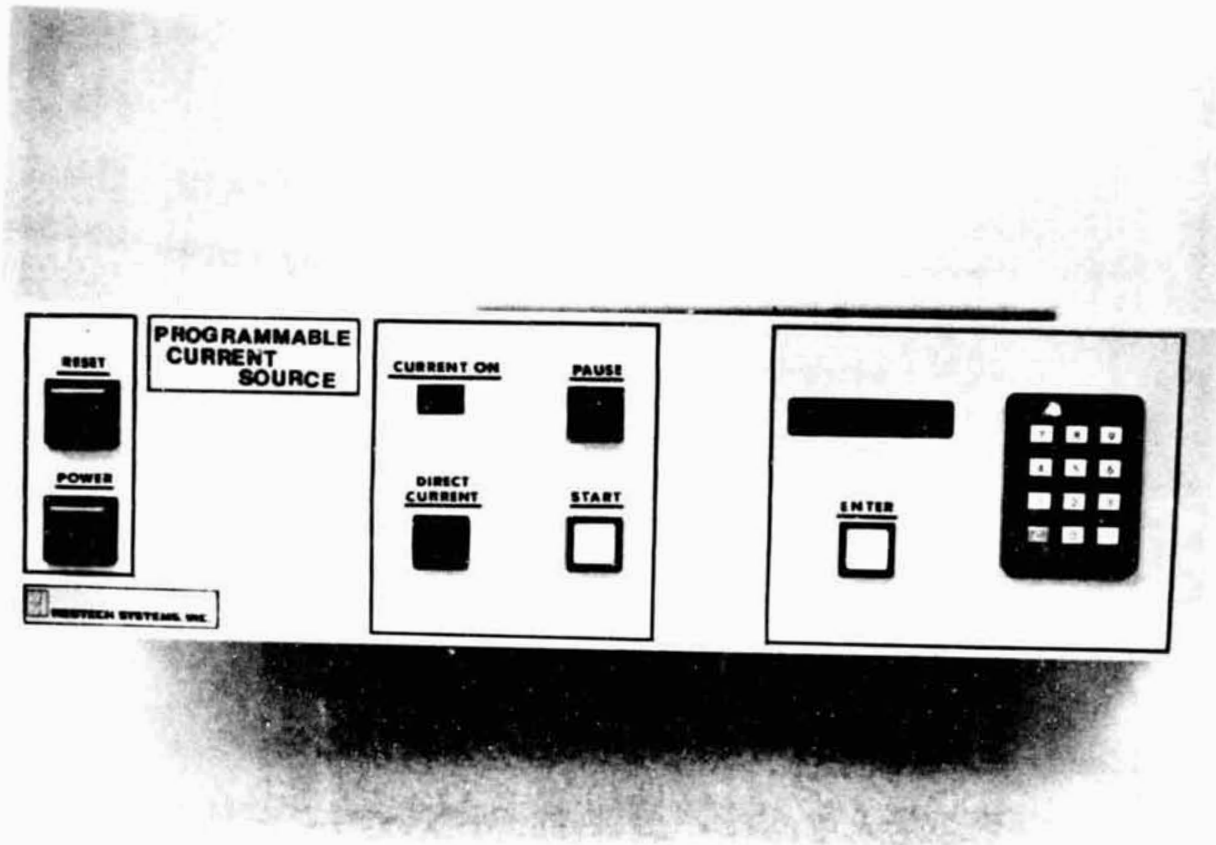


Figure 8. Peltier Pulser/Programmable Current Source
Front panel, with microprocessor on the right.

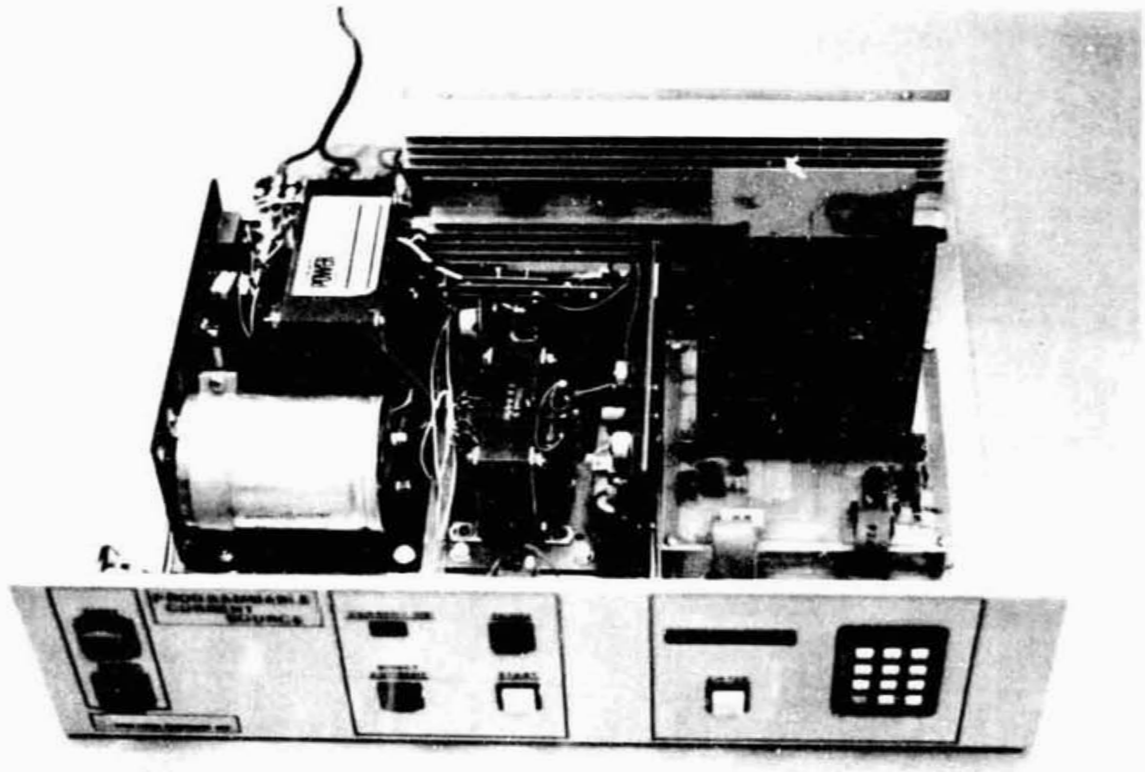


Figure 9. Chassis of Peltier Pulser
Power unit is on the left and rear
Microprocessor is at the right.

to microgravity experiments. Shuttle power would be used to charge Nicad batteries, which would then be used to generate the pulse. By a suitable protection circuit, the shuttle would not see the transient of the pulse.

C. MEASURING MELT AND SOLID TEMPERATURES

Ground based characterization of the silicon float zone (solid-melt-solid) configuration requires a knowledge of the thermal profile in the system. This will be used to understand heat flows within the solid. In turn, this will explain differences between the r.f. float zoning system and the Thin Rod Zoner configuration, which is resistance (hot wall) heated. In the r.f. configuration, the heat is concentrated in the melt and the melting front of the top (feed) rod. The length of the rods are not heated, and with fast loss of heat due to radiation along the solid rods, they cool rapidly in the r.f. method. The hot wall method heats the solid rods, resulting in lower temperature gradients along the rods. In addition to analyzing the ground based work on this contract, this heat flow information will be of use to Prof. Brown's analytical models (at M.I.T.) and Larry Foster's modeling of the hot wall furnace conditions and their effect on the growth interface shape (a contract with S.A.I.).

The first method of measuring temperatures was with a thermocouple inserted in a well along the axis of the feed rod during zoning (see Figure 10). As the zone approaches the thermocouple, the temperature increases. This is equivalent to moving a thermocouple far from the melting interface toward the melting interface at any given time. A W-Re thermocouple

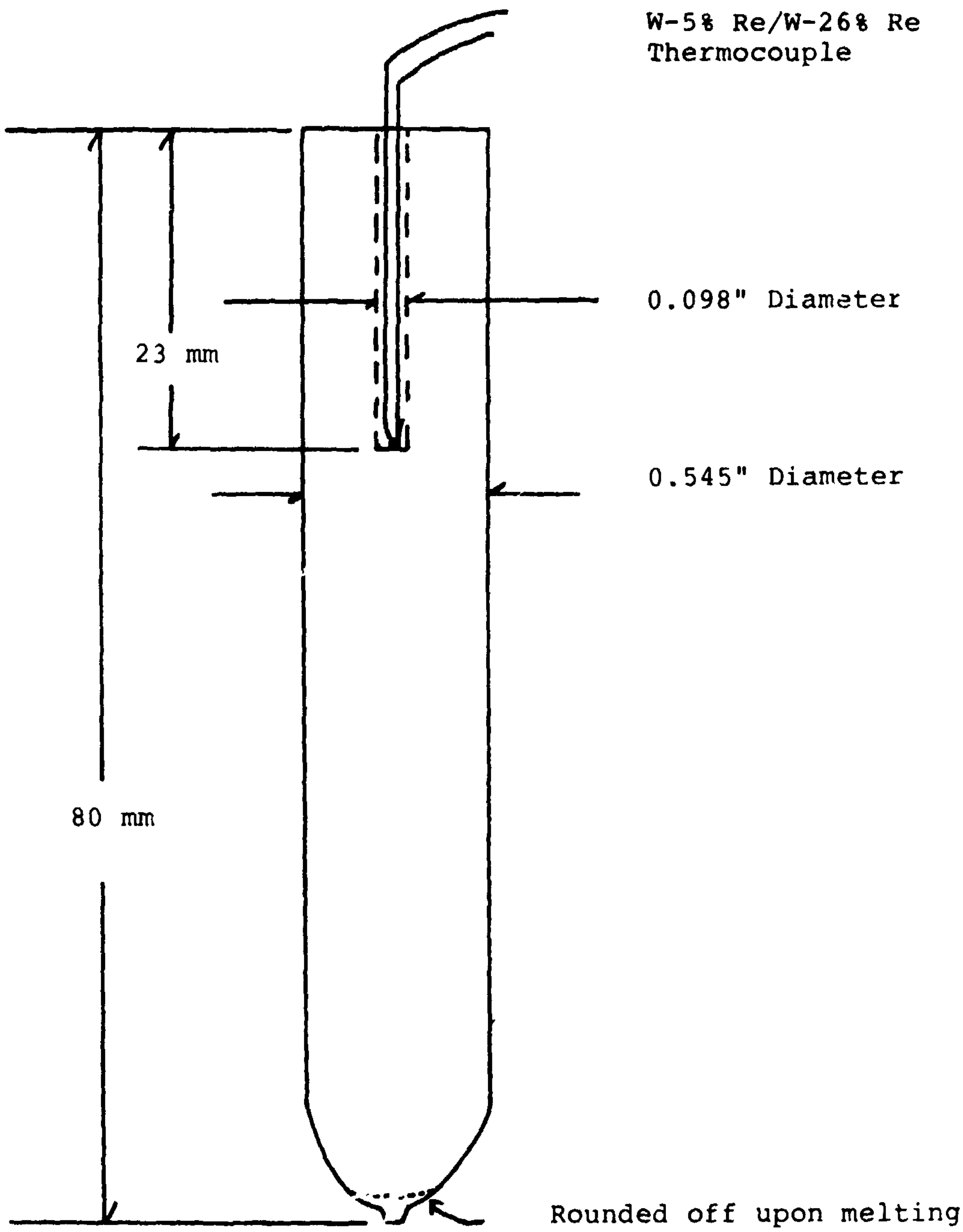


Figure 10. Profiling Silicon Rod Temperature with Thermocouple

was used. An axial cavity was machined out with a diamond drill. The thermocouple leads were brought out of the zoner and attached to the strip chart recorder. A 0.098 inch (2.5 mm) diameter axial hole was drilled 23 mm into a 80 mm long ingot. The start of the profile is 55 mm from the molten silicon interface.

The thermocouple was devised as a method to measure thermal profiles and transients in the melt, if a thermocouple could withstand the chemical attack from liquid silicon. The W-Re thermocouple used above was attacked very rapidly. While large magnitude and fast transients were observed on the recorder, we are not sure whether the thermocouple was intact during any portion of this time. It had been dissolved by the silicon after the run was completed and the thermocouple removed.

Pt/Pt 10% Rh thermocouples were also dissolved rapidly. Attempts by a glassblower to encase the thermocouple in a quartz protective bead and sheath were not successful. This requires a thin sheathing so that the heat capacity of a large quartz mass doesn't mask the fast thermal transient. An Al_2O_3 cement applied by dipping was too porous to provide protection. A sprayed BN coating has been recently applied to the thermocouples and will be tested in the beginning of the second year's effort.

A Barnes infra-red microscope was supplied by NASA. After delays in its original shipment, its missing the electronic control unit, and delays by Barnes Engineering in integrating that, the unit was finally delivered to Westech. The unit was subsequently mounted on the r.f. zoner (shown in Figure 11). Profiling was done on the solid-melt system, and the results are given in the next section. Since the Barnes microscope is built for a radiance

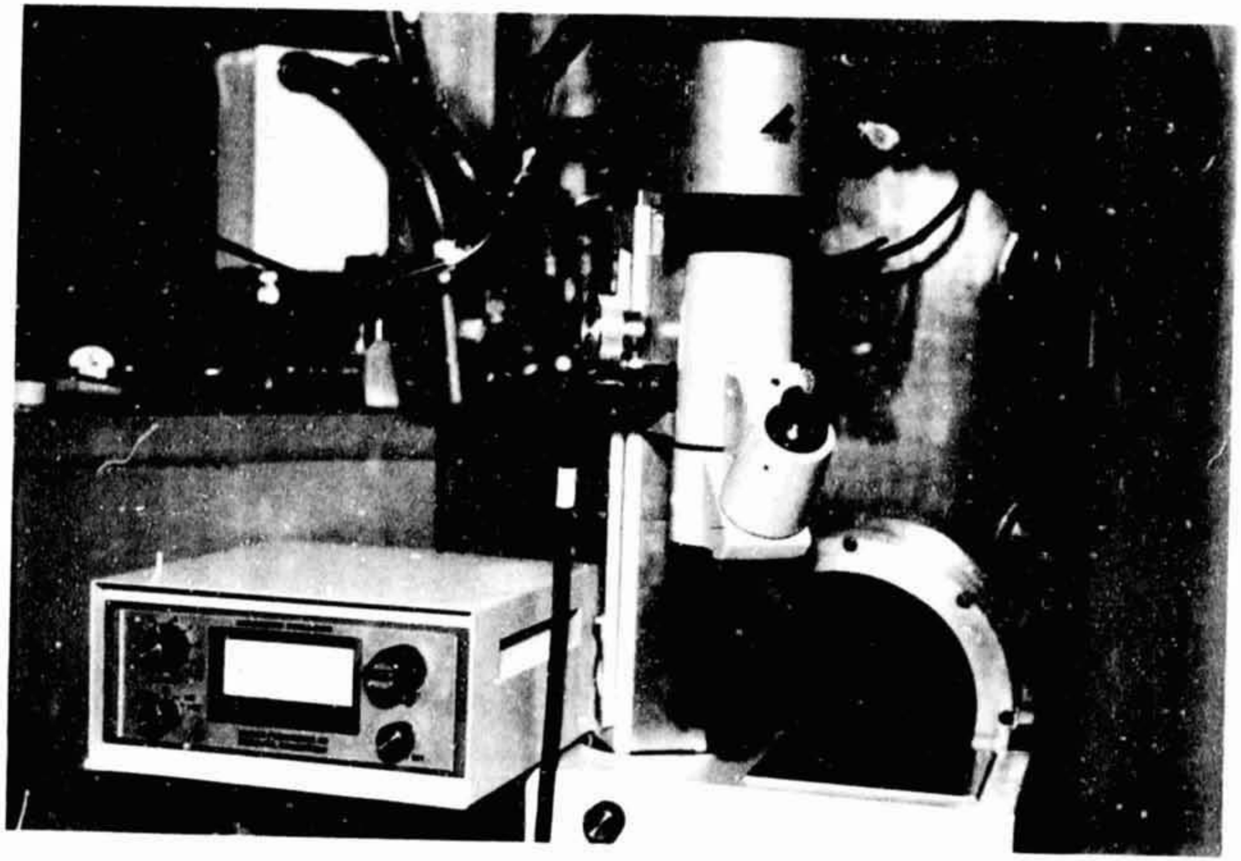


Figure 11. Barnes Infra-red Microscope mounted on Zoner
The objective is focused through the zoner window at bottom right. The new control unit is at the left.

scale maximum of 345°C, measuring of a silicon melt (1420°C) is done by stopping down an (iris) aperture within the microscope. Using the maximum reading on the solid silicon as a calibration point of 1420°C and the equations and radiance vs temperature curves in the Barnes manual and using a stated solid emissivity of 0.65, other temperatures are calculated from the chart recordings. The error by this procedure could be substantial. Calibration methods can be utilized if more precise values are really necessary, but these are difficult.

D. THERMAL PROFILES IN THE SILICON SOLID-MELT SYSTEM

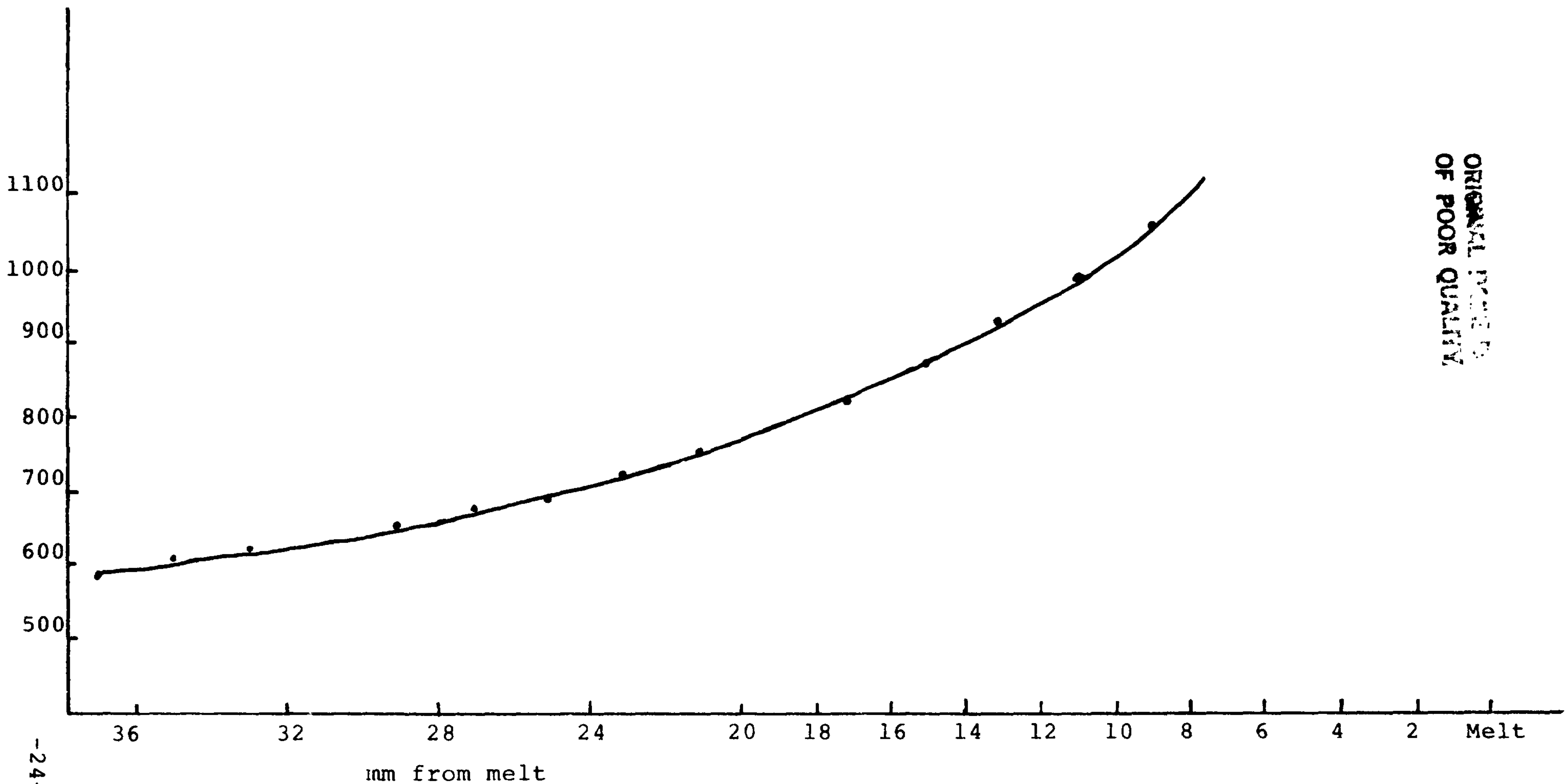
The axial temperature profile in the silicon solid, as measured by the thermocouple, is shown in Figure 12. The silicon single crystal diameter was 0.545 inches (1.38 cm) and the zoning speed was 2 mm/minute.

The axial profile in the silicon solid-melt-solid system, as measured by the Barnes I.R. microscope, is shown in Figure 13. The melt temperature was assumed to be 1420°C, since the large spot size and inaccuracies in the molten emissivity (usually taken as 0.35) lead to high inaccuracies. Note that the coil obscures most of the melt. The melt height was 0.56 inches (1.43 cm) and the coil width 0.39 inches (1.0 cm). Using the visible microscope part of the microscope and the melt-solid interface, the spot size (for the infrared) appears to be 0.065 inches (1.6 mm). The Barnes manual states 0.040 inches (1.0mm). Thus the melt height that can be viewed by the microscope is comparable to the viewing spot size.

Profiling of the melt with the I.R. microscope would be

Figure 12. Silicon Ingot Axial Thermal Profile

As measured with a W-5% Re/W-26% Re thermocouple. Temperature above 1050°C were not accurate due to zero suppression, for observing thermal oscillations.



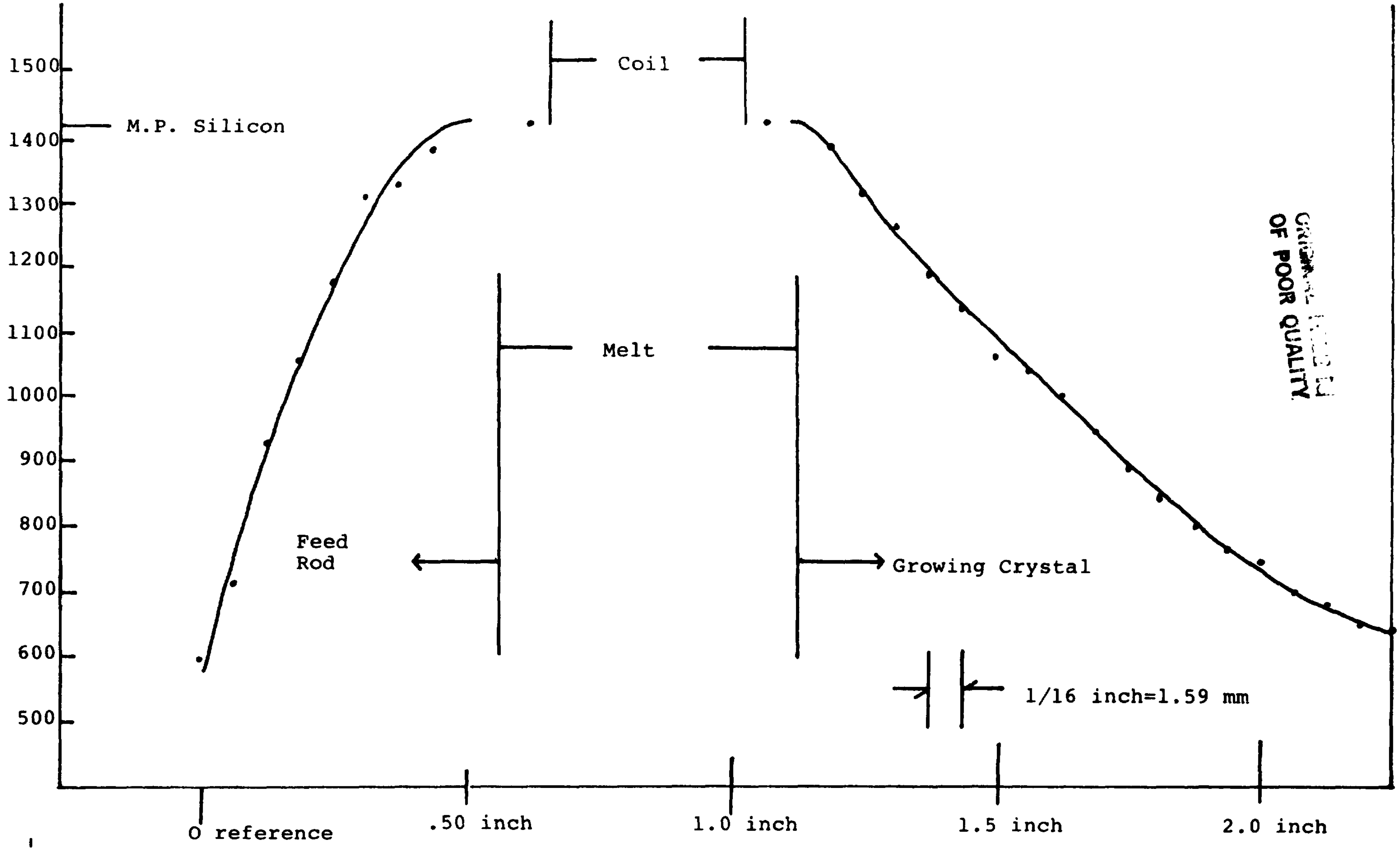


Figure 13. Profile of Silicon Solid/Melt Measured with Infra-red Microscope

possible if the coil obscuration is eliminated. This can be done by viewing the silicon system from an angle looking up, when below the coil, and looking down when above the coil. This can be done with a series of mirrors. Since radiation from a surface is a cosine function, the angle of viewing must be factored in. This modification and subsequent analysis will be done in the second year.

E. TRANSIENTS IN THE SILICON MELT

Thermal transients have been observed in (visible) transparent melts by Schwabe[2] and Chun and Wuest[3]. These are also thought to be the cause of the minor striations as revealed by etching[1] in the silicon float zone system. Direct observation of these transients and their correlation to striations would confirm this and relate a lot of striation data to the factors which influence them. Striation etching can easily analyze a lot of data from many ingots and different experimental conditions.

The thermocouple measurements are tenuous, as discussed in Section C above. These measurements were recorded on a Soltec Corp. model 12242 strip chart recorder, which has a response time of 0.03 seconds for a 1 inch pen deflection, which is much faster than the 0.25 second period for thermal oscillations previously determined[1]. By suppressing the zero of the recorder electrically, recording was done first on the 2 mv scale (0.2 mv = 16.6°C) and then on the 5 and 10 mv scales. When the thermocouple was still in the solid well, but close to the melt, temperature excursions of 30°C were observed, going to 50 and 65° just prior to when we think the melt contacted the

thermocouple. The time from minimum to maximum temperature was typically 1.5 sec., not unlike the striation etch striation periods (typically 0.25 to 1 sec.).

After the time when the thermocouple should have entered the melt, the temperature excursions were from 90° to 280° to 500°C, if the thermocouple potentials can be converted to temperature, which does not seem likely. The temperature excursions took typically 1-3 seconds.

This experiment needs to be repeated when a good protective coating has been found to resist molten silicon. It is hopeful that the present BN coating will prove useful.

Using the I.R. microscope with the crystal rotating, periodic variations correspond to 3 times the rotation rate and are probably due to the <111> growth lines. The rotation was stopped and periodic fluctuations of from 1.5 to 4.2 seconds are observed. Since the vertical position of the interface is fluctuating at this rate, as observed in the visible eyepiece, this fluctuation is affecting the instantaneous growth rate. Since the I.R. measurement is seeing a fluctuating amount of solid and melt, with different emissivities, we cannot infer a temperature change from the amplitude.

F. SLICE ZONING EXPERIMENTS

Observing the influence of Marangoni flow in a high surface-to-volume configuration was suggested by J. Verhoeven in 1981[4]. This can be done by melting the center portion of a thin silicon slice and refreezing (crystal growth) at a controlled rate. Preliminary experimentation was reported in the last report (ref. 1, p. 46). This year, the electron beam melting experimentation by I. Dalins has continued at MSFC.

Gallium doped slices were provided from this contract. 1.2 inch diameter silicon rods were grown with a Ga concentration of 10^{16} atoms/cm^3. The crystals were sliced, etched and polished[1] to a thickness of 25 mils (0.62mm), as was desired for the melting experiment.

The slices were mounted in a ring shaped holder (at MSFC) for melting in the electron beam vacuum unit. This ring provides an electrical ground and a heat sink to the slice. A screen structure was built by I. Dalins to reduce space charge effects. The schematic of this configuration is shown in Figure 13. To melt the center of a slice, the voltage on the electron gun is manually increased. Initial attempts resulted in quite a few slices that were heated too rapidly, resulting in the melt collapsing (or blowing) and creating a hole in the slice. Careful control led to elliptical regions of melt up to 17mm in the long axis. The regrowth (recrystallization) was done by turning the voltage down. Rapid cutoff leads to very polycrystalline structure. The need for an electrically controlled ramping of the voltage (power) is evident. At this stage, the electron beam work was terminated at MSFC due to the transfer of Mr. Dalins

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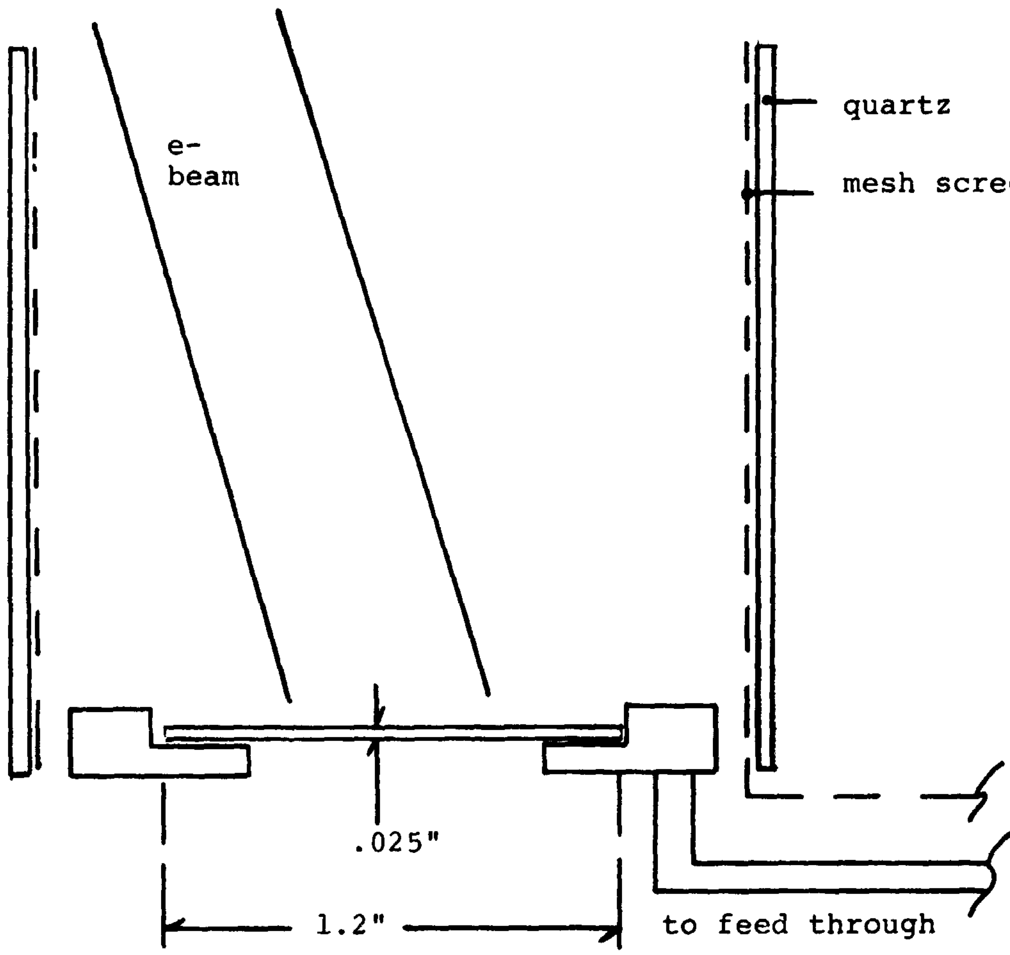

Figure 14. Electron Beam Melting of Silicon Slice

to another laboratory.

The recrystalized samples are cross-sectioned for analysis, and this was done at Westech. The slices are first encapsulated in epoxy and mounted on a silicon block for slicing through the cross-section. The epoxy provides rigidity to the very fragile silicon regrown slice. The silicon block is a rigid mounting for slicing and polishing and provides a flat means of holding the slice on edge on the microscope stage. After polishing, the slice is striation etched and the slice observed by Nomarski microscopy.

The shape of the recrystallized slice is roughly as expected. Figure 15 shows a cross-section. The bottom of the slice is next to the silicon mounting block. The top of the regrown slice is bulged more than the bottom. This is probably because the electron beam heats from the top only. The bottom, which is losing heat by radiation, will be the first to solidify, leaving the top to do the 9% volume expansion upon freezing[5]. Figure 16 shows that the trijunction angle is the 11° reported by Surek[6]. This is the angle between the solid, the liquid silicon (as observed after it has frozen) and the vacuum. This is most easily seen at the location where the last portion to be melted was in contact with the flat, polished silicon solid.

The problem with rapid solidification is shown in Figure 17. The rapid, uncontrolled-rate solidification leads to formation of polycrystal regions. If striations due to melt flow are present, they would be masked by the grain boundaries. The regrown slice shown in Figure 15 did show striations in portions of

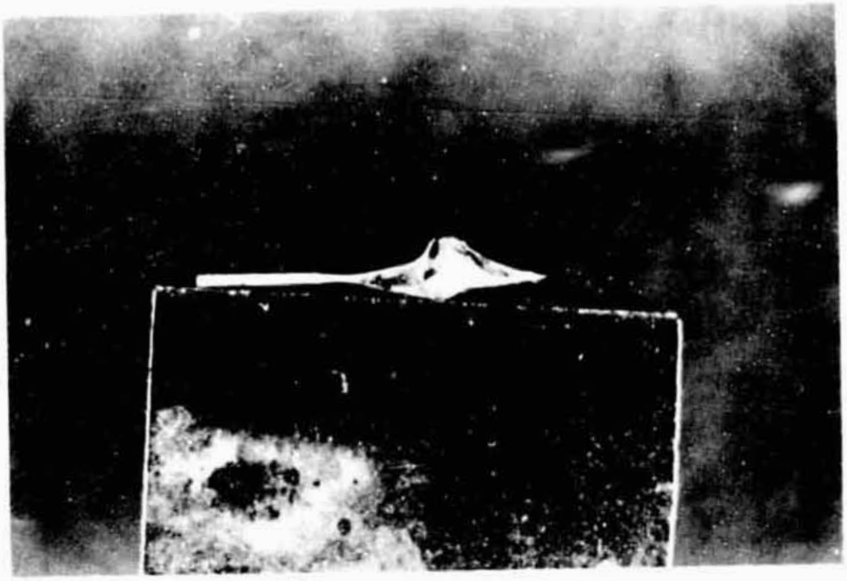

Figure 15. Cross section of Regrown Slice
Note original slice thickness at left.

Figure 16. Trijunction Angle of 11°
Original slice surface is at the left and resolidified melt at the right.

the slice. Many regions of the cross-section have numerous small grains, slip and a lot of defects and do not show striations. These effects may or may not lead to dampening melt flow and preventing the oscillatory variations which are observed as striations. To understand the striations and what causes them, it is essential that the regrown slices be single crystal and that the growth rate is controlled and known.

Figure 18a (at 200 magnification) shows the striations in an interior part of the slice; a large grain. The striations do not extend through two narrow grains, the outer one of which is the slice surface. This could mean that the phenomenon is a volume flow and not a surface flow, and therefore a buoyancy flow. It will also be instructive to compare this e-beam melted and regrown slice to a radiantly heated regrown slice. Individual vacancies can be seen, as well as vacancies clustered into a line.

Figure 18b (100 magnification) shows the periodicity of the striations on the opposite side of the slice. Unlike Figure 18a, these striations are roughly parallel to the surface, but again do not extend to the surface. Whereas the light striation lines are very regular, there are heavier lines (every 4 to 10 divisions and 1 division = 11.4 μm) that are aperiodic. This would suggest an aperiodic fluctuation superimposed on the periodic fluctuation. Vacancies are clustered on some of the major striations.

Figure 18c (200 magnification) shows another part of the same grain as Figure 18a. This slice was regrown when the electron beam was turned down rapidly. This could result in a growth

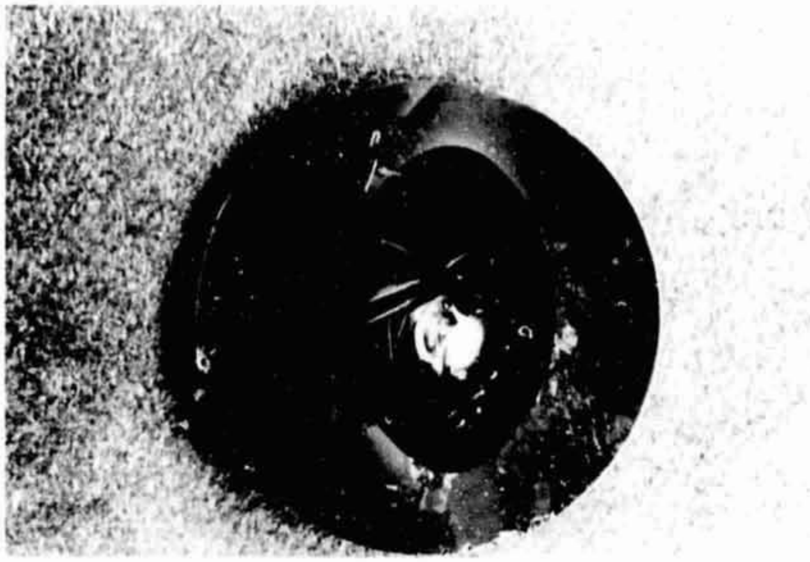

Figure 17. 17mm long melt zone.
Fast solidification led to polycrystal regions near center of regrown region.

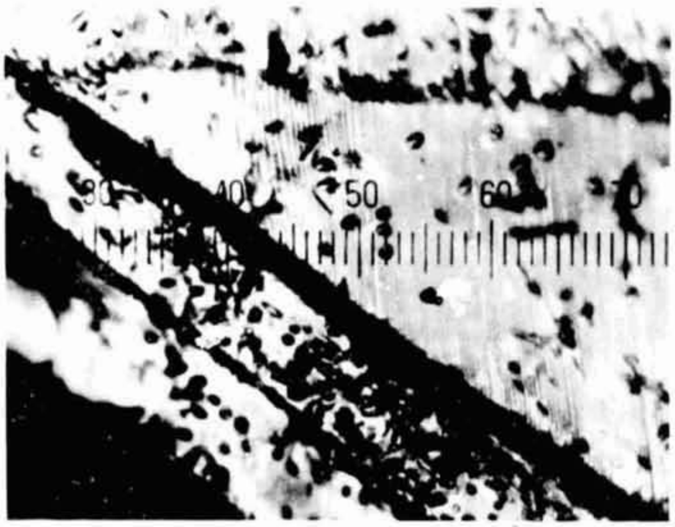

Figure 18a. Striations at Interior of Regrown Slice.
200 x magnification. The slice surface is at the lower left.

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Figure 18b. Striations of Regrown Slice (100 x magnification). Shows aperiodic heavy lines (ex. at lines 41, 55 and 63).

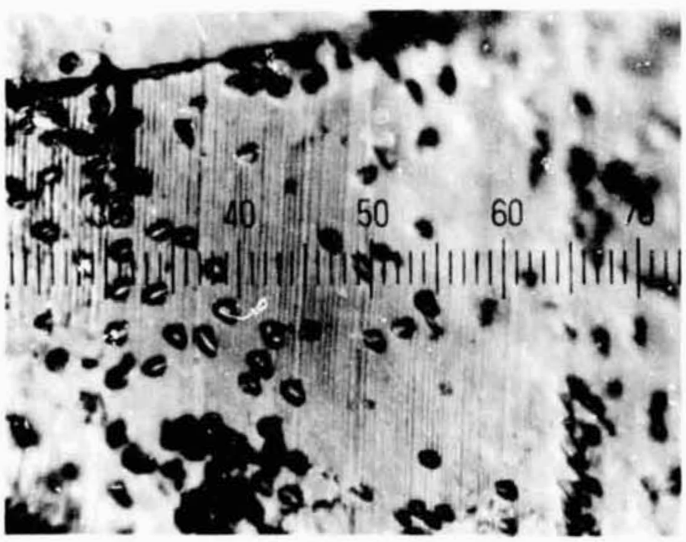

Figure 18c. Regularity of Striations in Regrown Slice (200 x magnification)

rate of about 1mm/sec. Since each striation is about 1/2 a division, or 2 μm, the period would be about 2 msec. Heavier striations appear about every 10 divisions (50 μm or 0.05 sec.). These compare to 1 inch diameter zoned rods with a striation period of 0.25 to 0.5 sec.

It is recommended that a small experimental vacuum chamber be constructed, using elliptical heaters. This can be easily controlled for heating and cooling rates. Such a system will be easy to fabricate, control and operate. Gases can easily be introduced into this system. Radiant heating would also eliminate the question of any possible effects by electromagnetic pressures.

An easy method to observe whether rapid stirring has occurred in the melt is to dope one surface of the solid slice, then rapidly melt and solidify and profile the slice cross-section for resistivity. A very shallow doping can be don controlled manner using ion implantation. Gallium is not a common ion implantation source; so boron can be used. This ion implanting can be done in the Arizona State University semiconductor technology laboratories.

The coating of a liquid surface with a solid will eliminate Marangoni flow. The coating could be extremely thin and could probably have cracks and some voids, as long as it covers most of the surface. Mixing in an r.f. heated zone is known to break up SiO_2 coatings. If the silicon growth goes from the outside surface to the inside, as with r.f. heating, incorporation of SiO_2 at the growth interface leads to polycrystal growth. If, however, the outside is the last to freeze, as with a convex solidification interface, stresses on the outer boundary do not

lead to polycrystal growth, as observed with the vapor growth of CdTe[8].

Regrowth of polycystal silicon layers into epitaxial single crystal layers on top of a single crystal substrate, while encased in solid SiO_2[9,10] leads to the possibility of putting a solid SiO_2 or Si_3N_4 layer on the outside of a silicon melt and still produce a single crystal, especially if the growth interface is either flat or slightly convex.

A new slice zoner should have the ability of in-situ growth of SiO_2 by a controlled vapor pressure of CO_2 (a low energy source of reactive oxygen) or of Si_3N_4 by a controlled vapor pressure of NH_3.

II. TASK 2: COMPARISON OF ZONE HEATING METHODS

INTRODUCTION AND SUMMARY

The objective of this task is to determine the best method of heating silicon to obtain molten zone characteristics which will lead to more uniform crystal growth. The major benefit expected by float zone growth in microgravity for silicon and other technically important semiconductors is more uniformity in resistivity, recombination lifetime and optical properties and a lower and uniform density. Microgravity is expected to minimize flows within the molten zone. The heating method selected should minimize disturbances in the melt and at the growing crystal interface.

The goal of this task is to select the heating method to be used in early flight experiments in float zoning of silicon. This is now projected for a flight as early as 1986, with the experimental equipment to be housed in an EAC (Experimental Apparatus Container) to be mounted on the Hitchhiker carrier (MPS carrier for experiments) in the Shuttle bay. Considerations need to be given to a) total power requirement and power efficiencies, b) voltage requirements, c) size and weight, d) environmental effects on the Shuttle (such as EMI) and e) the amount of development work required. Comparison of the various heating methods is reported in section A below.

R.F. zoning, which is the commercial process to zone refine silicon for stringent power and (optical and radiation) detector devices, would need improvement in several areas to be considered

for early microgravity experiments. Section B, below, describes concept work to improve power efficiency. This effort was terminated part way through the contract year, when the heating method selection was made.

The heating method selected for early flight experiments is the resistance heated-hot wall method, which was developed under Task 3 of this contract. It is recommended that this furnace design be optimized for zoning silicon at 5 mm diameter and that additional ground based work characterize the zoning process in this equipment and the crystal grown.

A. COMPARISON OF ALTERNATIVE HEATING METHODS

Alternative heating methods were discussed in the original proposal for this contract ("Microgravity Silicon Zoning Investigation" E.L. Kern, G.L. Gill and O. Stafsudd, Westech Systems, Inc. submitted to MSFC Dec. 18, 1981) for an experimental furnace proposed for the M.E.A. carrier. Laser melting was not considered due to its very poor power efficiency (<1%). The other methods are reviewed below, with discussion including the five concerns mentioned in the above Introduction section, as well as the ability to tailor heating profiles and the mixing effect inherent with the process.

<u>R.F.</u>: Zoning with a high frequency inducing eddy current heating into silicon was the method used to purify silicon, which led to its use in all silicon devices [11, 12]. Since then, it has been used to make very high purity silicon for power devices and for infra-red and visible imaging arrays important to the military and space programs. The technology for commercial zoning,

has been well developed[13]. The key concerns which need to be addressed for microgravity zoning are:

- Power efficiency: Commercial units with single turn r.f. coils are currently about 20% efficient. Earlier Siemens' zoners were as high as 33%. Improvement to 50% is envisioned, but would require a major development effort.
- Cooling Requirement: Power lost in producing r.f. power and heating of the chamber walls requires cooling - approximately 75% of total power used.
- Voltage: Commercial zoners transform 480 v.a.c. up and rectify to obtain 13 kv d.c. The transforming and rectifying are inefficient processes.
- Size: Air gap voltage isolation and complexity of power supply will require additional space.
- Weight: Present power supply methods are very heavy, but solid state power supplies will be considerably lighter. Still requires more power supply than resistance heating.
- Environmental Effects: High voltage/high frequency leads to more EMI which would have to be filtered and shielded, requiring more weight and space.
- Zoning Atmosphere: Can zone in high vacuum ($\leq 10^{-4}$ torr usually) or inert gas (1 atm. Argon). Discharges occur : 10^{-1} to 10^{2} torr.

- **Heating Profiles:** Heating is concentrated in a narrow ring in the molten zone. Methods for heating longer zones(12) or putting heat into the solid rods have been tried, but have not resulted in successful processes. R.F. heating leads to local overheating, maybe as high as 25 to 50°C, which will drive Marangoni flow. The necessary split in the r.f. work coil also leads to major striations (rotational meltback) which gives high resistivity variations and a high density of swirl defects.
- **Melt Mixing:** R.F. frequencies (2.4 mhz) are predicted to give a large amount of melt mixing (14). How this combines with buoyancy and Marangoni flows to give rise to aperiodic striations is not known.
- **Stage of Development:** R.F. zoning is developed to a stage of reliable production equipment and a repeatable industrial process, with a long history of successful material usage.

R.F. float zoning is the reliable, technically proven and commercially important technology to use as a basis of comparison. It has inherent problems in producing the superior property crystal that is desired for advanced state-of-the-art devices for critical sensing array technologies. Its translation to microgravity processing would entail a large development effort and would

result in at least moderate weight, size and power efficiency penalties.

Electron Beam: Electron beam zoning is used routinely to purify high temperature metals, such as a tungsten and molybdenum used as a sputtering source for silicon device fabrication[12]. Silicon has been successfully zoned, with the process being developed just up to the point of going into production[16].

∘	Power Efficiency:	The d.c. power generated by the filament is high with losses incurred by the filament heating, losses in converting to high voltage and due to directing of the beam. If the beam has to be extracted into a gas atmosphere, further losses are incurred. The efficiency is 30-50%.
∘	Cooling Requirement:	Inefficiency in power supply must be carried away by coolant. Chamber must be cooled. Approximately 75% of power used must be cooled.
∘	Voltage:	Several kv.d.c. The transforming and rectifying are inefficient processes.
∘	Size:	Power supply is bulky. Added length usually needed to keep electron gun from contaminating the melt or to extract beam from a vacuum into a gas atmosphere.
∘	Weight:	Power supply and high vacuum pumping will add weight.
∘	Environmental Effects:	High voltage would require EMI filtering in the power lines.

- Zoning Atmosphere: Easy to zone in high vacuum, but requires added feature so electron gun does not contaminate the melt. To zone in an inert gas, the beam must be extracted into the gas chamber, requiring differential vacuum pumping, which adds power, weight and size.
- Heating Profile: Electrons can be focused with potential fields. Heating over a long length would require multiple guns, especially to have the flexibility seen in resistance heating.
- Melt mixing: Some mixing is expected due to a concentrated beam (creating a psuedo pressure).

Electron Beam zoning has some of the disadvantages that r.f. zoning has (high voltage, power inefficiency, size and weight penalties). It does not have a line discontinuity that would give rise to major striations (unhomogeneity). Zoning in a gas would be inefficient and require considerable development.

Radiant Heating: Lamps focused by elliptical reflectors have been used to zone refine germanium in the past[12]. More recent work by Eyre and Nitsche[17] have led to flight experiments for zoning silicon in a double ellipse furnace in Spacelab (Oct. 1983). More recently, silicon has been melted in a monellipse furnace[18]. The monoellipse furnace is being built to fly in Spacelab in 1984 or 1985, to do limited zoning within a quartz ampoule and Bridgman growth.

The monoellipse furnace has an azimuthly uniform heating

zone, giving it a distinct advantage over the nonuniform heating pattern of the double ellipse, which leads to thermal gradients in the silicon zone of 15°C[19]. The monoellipse can melt 5mm length of 5mm diameter silicon with 300 watts. This compares to 88 watts to melt silicon in the Thin Rod Zoner (Section III-D below) under 10^{-1} torr vacuum and 170 watts at 1 atmosphere (Argon). The monoellipse furnace is not designed for either gas or vacuum zoning and does not have a movement method developed. Development of these features looks feasible, but would require considerable development.

° Power Efficiency	The inefficiency of resistance heating a tungsten lamp, absorption by the elliptical reflector (10% of incident energy at best) and losses due to holes in the reflector lead to an efficiency of about 30-40%.
° Cooling Requirements:	Lamp, sample and elliptical reflector must be cooled, for about 90% of the total power used.
° Voltage:	About 100v, requiring step-up from shuttle supply, but much more efficient than r.f. and electron beam.
° Size:	Reflector about 30 cm diameter x 35 cm long. Larger than resistance heated furnace.
° Weight:	No heavy power supplies. Moderately light.
° Environmental Effects:	No problems with EMI. No high voltage.
° Zoning Atmosphere:	Could zone in gas, but with heavy heat losses to the elliptical

		reflector. Moderate vacuum (down to 10^{-2} torr) is good. High vacuum would lead to coating the reflector, which cannot be done.
°	Heating Profiles:	Eyre[18] showed some control of a thermal profile along a sample by moving a heat shield. More profiling is possible than for r.f. and electron beam, but less than radiant heating.
°	Melt Mixing:	Very little.
°	Stage of Development:	Only initial research done on melting silicon in a quartz ampoule. Zoning would require extensive development.

Radiant heating in a monellipse furnace is plausible, but would require extensive development. A double ellipse furnace will be run with silicon on Spacelab, but the thermal inhomogeneity is expected to defeat the goal of a very uniform crystal. These results should be reviewed.

Radiant heating appears to have the disadvantages of not being the most power efficient process and has limited thermal profiling ability.

<u>Resistance Heating</u>: Float zoning with a resistance heated furnace for high temperature semiconductors has not been done. Zone refining of lower temperature materials (<500°C) in quartz or graphite boats and tubes is done routinely. When compared to r.f. zoning of silicon, several areas are of immediate concern: a) the operator cannot see the melt or growing crystal interface, b) the length of melt that can be supported without levitation, c) whether a sharp enough temperature maximum peak

can be made for the length of zone desired and d) how much heat needs to be extracted from the bottom and top rods.

The resistance heated furnace designed by R. Mellen and operated by Dave Larsen (Grumman Aircraft) was reviewed and observed in initial operation. This approach, of many axial segments to make up the profile, and the fact they can be separately controlled, provides a lot of flexibility to creating a given thermal profile. The large dimensions of the furnace, which is composed of large circular rings, and the large number of power supplies (one of each axial segment that is set at a different current), does not seem to make the furnace easily adapted to early flight experiments.

The ADSF (Advanced Directional Solidification Furnace) made by G.E. and flown on SPAR flights is a compact and light furnace successfully used for Bridgman growth of metal samples. Its performance was reviewed with Dave Larsen (Grumman) and Mary Helen Johnston (MSFC Materials and Processing Lab). Some components for one furnace were obtained for aiding in design and fabrication. If the thermal profile can be adapted to the float zone requirement, this furnace appears suitable for early flights.

Further flexibility is afforded by a variety of ways of resistance heating to create a desired thermal profile. These include:

a. Precious metals (Pt,Pt/Rh, etc.) or refractory metals (W, Mo, Ta) variably wound in a ceramic (AL_2O_3) muffle tube,

b. Graphite tube, which can have a variable wall

thickness to control temperature,

c. ZrO tube (Artcore, Inc.), which can have a variable wall thickness to control temperature.

The key comparative concerns are:

° Power Efficiency:	As high as 70%, depending upon the efficiency of insulation and need for gas flows or preheaters and the aspect ratio (diameter/length) of the muffle tube.
° Cooling Requirements:	Can be low, if insulation is very good. If power efficiency is very high, about 75% of power would need to be extracted by cooling.
° Voltage:	Low voltage. $\leq$ 24 v.d.c. is compatible with direct heating with Shuttle power without the inefficiencies of power conversion.
° Size:	Small. ADSF type furnace is 6 inch long x 4 or 5 inch overall diameter.
° Weight:	Low. ADSF type furnace weighs 4 to 6 pounds. No heavy power supplies.
° Environmental Effects:	No problems with EMI. No high voltage.
° Zoning Atmosphere:	Either vacuum or inert pressure possible. Pt heating elements sublime $\leq 10^{-3}$ torr, so higher vacuum would require a vacuum pumped tube for the sample.
° Heating Profile:	High degree of flexibility, using such designs as variably spaced

		and multiple layer windings and tickler coils.
°	Melt Mixing:	None.
°	Stage of Development:	Developed as a laboratory furnace for Bridgman growth and tube zone refining, but not known for float zone growth.

Resistance heating (hot wall with profiled temperatures) looks promising for early flight experiments for silicon. The lack of development for float zoning will require that a considerable amount of research be done. No large equipment developments, such as complex power unit miniaturization, are required. There is a large amount of knowledge for resistance heated furnace tubes, used in laboratories and semiconductor production, to be drawn on.

<u>Recommendation</u>: The most promising method, from the comparative advantages of:

a) High power efficiency and low cooling needs

b) Small weight and size

c) Use of shuttle voltage directly

d) Tailoring the thermal profile to grow crystals with better properties

is the resistance heating method. An adaption of the ADSF design should be tried first.

The results of microgravity flights of the double ellipse furnace and monoellipse furnace should be reviewed. Experiments of melting silicon in the monoellipse furnace should be explored with the German (ESA) microgravity material processing efforts.

B. CONCEPTUAL IMPROVEMENT IN R.F. HEATING

R.F. heating is the commercial method for growing silicon by the float zone method, which leads to process and equipment reliability and at least understanding of how to use the process. It would, therefore, be desirable to also use this process in space, if it were not for two distinct disadvantages. The r.f. coil has a slot to prevent shorting of the r.f. circuit and this creates a major striation, with corresponding variations in electrical and crystallographic properties. Attempts in eliminating the slot last year[1] did not lead to promising results. The other problem, low power efficiency, was looked at this year.

Efficiency of power application and efficient utilization of space available for the coil and power transformation is a concern. A working prototype combination R.F. coil and impedance matching transformer was built to test the feasibility of this configuration and is shown in Fugure 19. This is a concept similar to one used to concentrate power into a short coil.[15]. The unit was installed in the breadboard zoner with the R.F. oscillator connected to the outer coil. Teflon insulation surrounds the one turn secondary which is in turn connected to the work coil within. Both the secondary and work coil are split. Cooling water is circulated through both the secondary and work coil and is connected to the chamber ground from a position opposite the coil slot as is done in standard R.F. zoning.

A single functional test was completed with an 18 mm silicon rod being zoned 4 inches in length. Although the run was not instrumented for power measurement, the rod was run with about 20% less power setting than that used for a similar sized rod

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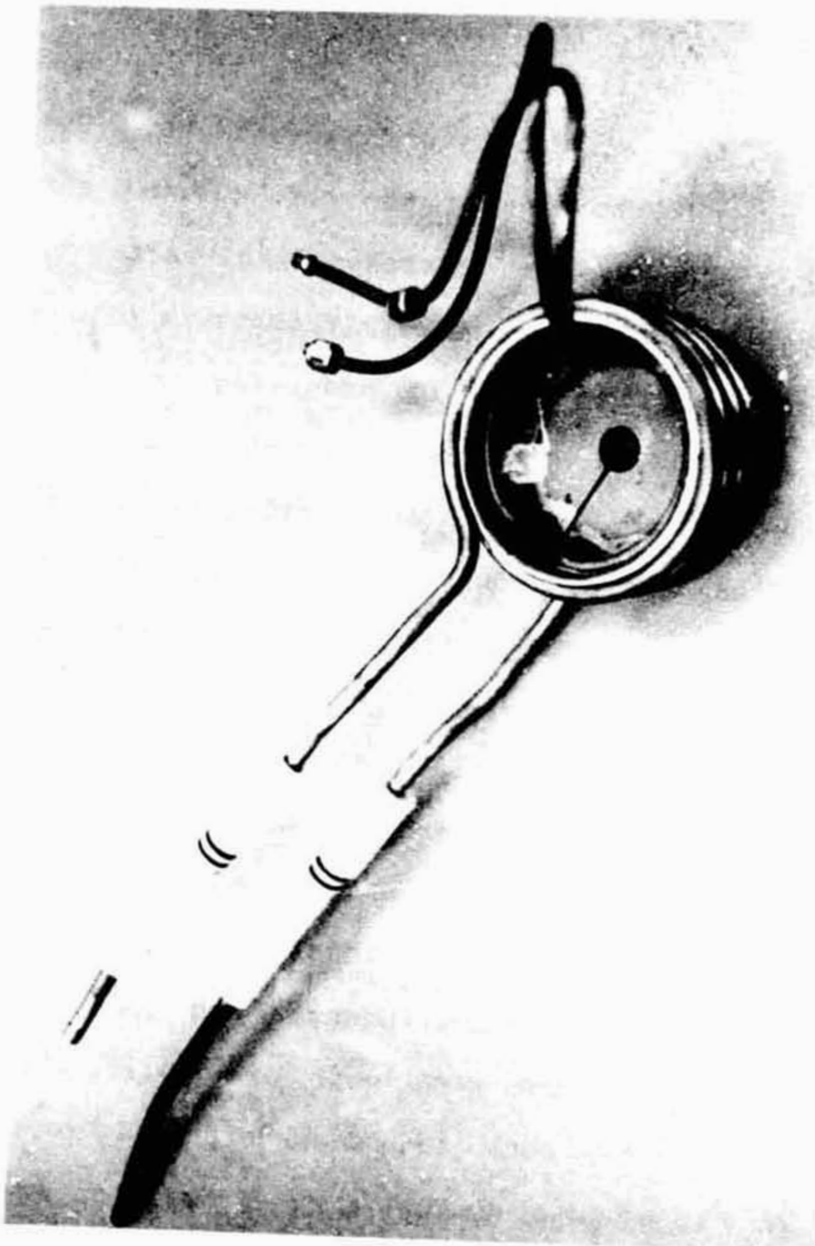


Figure 19. R.F. Experimental Transformer/Coil
Lines at left feed power in and cool primary transformer coil.
Water lines at right cool secondary/work coil.

using the "standard" configuration. This demonstrates that power efficiencies can be improved. (This year's contract proposal called for a needed 50% power efficiency.)

Another method to eliminate the effect of the coil slot is to heat a round secondary heater. Since ZrO_2 is conductive at high temperatures, its use as a heater was tried. A high density, high purity ZrO_2 cylindrical sleeve (0.75 inch O.D., 0.10 inch wall thickness and 0.8 inch long) was placed on top of a r.f. zoning coil, with a silicon rod inside and a graphite suseptor block below. The graphite heated the silicon until it was conductive, which in turn heated the ZrO_2. The latter reflected heat back into the silicon, which was zoned with less power than normal. The grown crystal was slabbed and etched, revealing the normal rotational meltback striations. It appears that the r.f. field goes through the ZrO_2 and still provides the primary heating of the silicon. The ZrO_2 acts as a heat reflector and, maybe, as a secondary heater. This is not a method of eliminating primary striations.

When the resistance heating method was selected for early flight experiments (Section A above) and reviewed (23 March, 1983 Float Zone Working Group meeting at MSFC), this effort on r.f. improvements was terminated and emphasis put on the Thin Rod Zoner.

III. TASK 3:

CHARACTERIZE THE FLOAT ZONE IN SMALL DIAMETER SILICON RODS

INTRODUCTION AND SUMMARY

The objective of this effort is to float zone silicon as it might be best done in early flight experiments and to determine the characteristics of this type of zoning. All commercial float zoning of silicon is done with r.f. (radio frequency) heating at about 2.5 mhz. This is not practical in space for three reasons: a) an r.f. power supply is usually large and customizing it to a space vehicle would be difficult and expensive, b) an r.f. power supply and subsequent coupling of power into the silicon melt is not energy efficient, with present zoning efficiencies being between 20% and 33% (at best) and c) the split coil necessary for zoning introduces rotational meltback striations which is a major inhomogeneity we are attempting to eliminate. It is therefore desirable to go to either a resistance heated method or a radiant heated method. The NASA ADSF furnace provides a model for resistance heating, although the thermal profile would have to be totally redesigned.

Early flight experiments will require that the zoner equipment go into one of the small equipment carriers being used for materials processing. Since zone refining takes equipment that is 30-36 inches in length for zoning lengths of ingots that give suitable data and since cooling is involved, it is considered inappropriate for the shuttle mid-deck. A carrier in the shuttle bay, such as MEA or the Hitchhiker is appropriate for power and cooling levels and size and weight. The furnace fabricated on

this effort is designed to be used in an EAC (Experiment Apparatus Cannister) in such a carrier, with sizes and power being compatible to a MEA or Hitchhiker carrier.

The goal of this year's effort was to design, build, debug and operate a Thin Rod (resistance heated) furnace for zoning silicon. The zoning operation should be characterized to show the power and cooling required from the shuttle carrier. While it was desired that commercial heaters be used, these were found unsuitable during the startup of the furnace and development of heaters was required to keep the program viable. Except for the electronic controls and power supplies, the furnace is a form and function prototype of a flight furnace.

A. DESIGN AND FABRICATION OF THE THIN ROD FURNACE

The Thin Rod Zoner design followed several design precepts based on successful small furnaces: a) the heater design was patterned after the ADSF furnace, b) the mechanical movements were based on scaling down successful designs used in commercial zoning. The heater design will be discussed in Section B below. The assembled zoner is shown in Figure 20.

The cylindrical chamber in the center of Figure 21 is the atmospheric chamber for zoning. This is the only part of the zoner where a controlled atmosphere for zoning is necessary. The open vacuum flange, at its center, is for attaching a vacuum pump, if high vacuum is to be ued, such as a small vac-ion pump. Otherwise, forepump vacuum and gas atmospheres can be introduced at this point. Forepump pressure (to 10^{-2} torr) can be provided by piping to the shuttle bay. In order to minimize the length

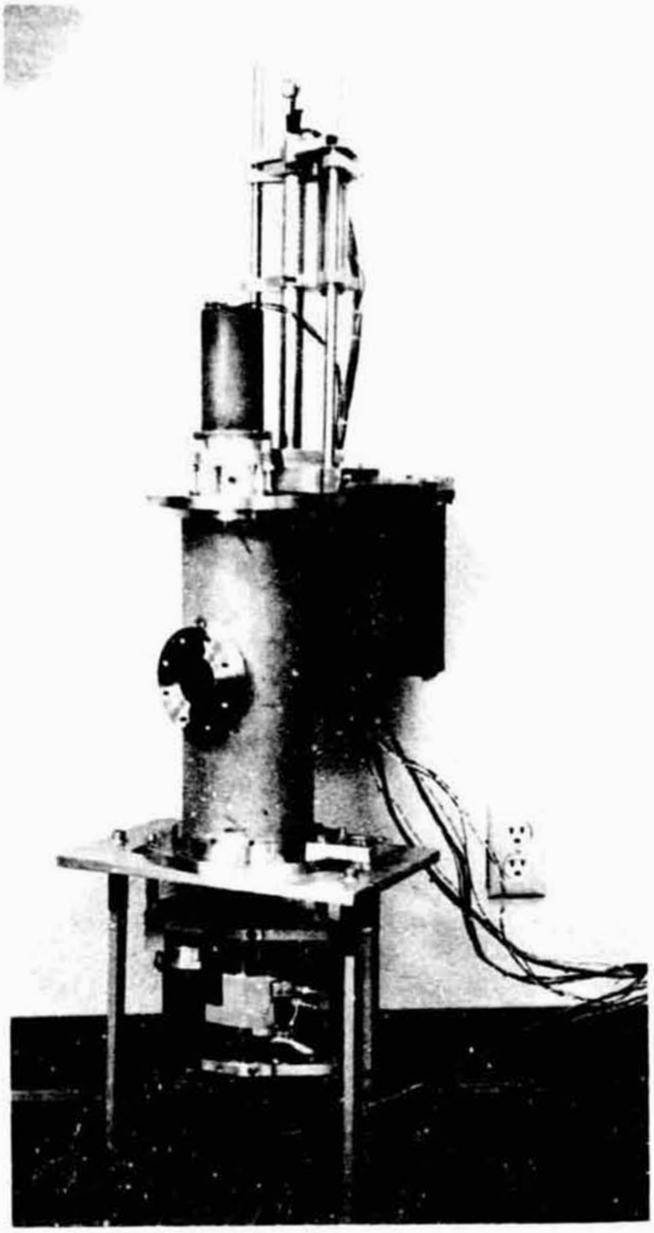
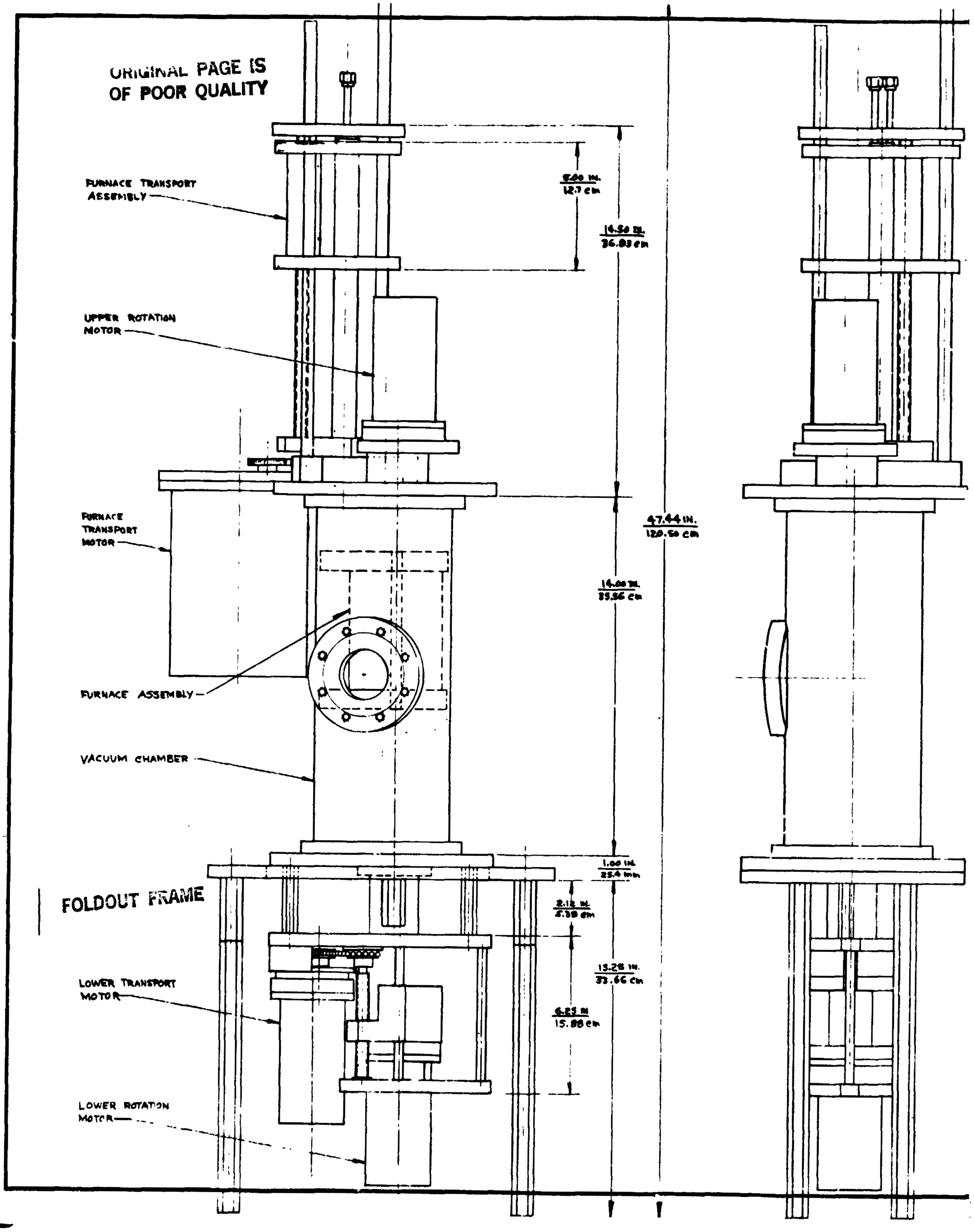
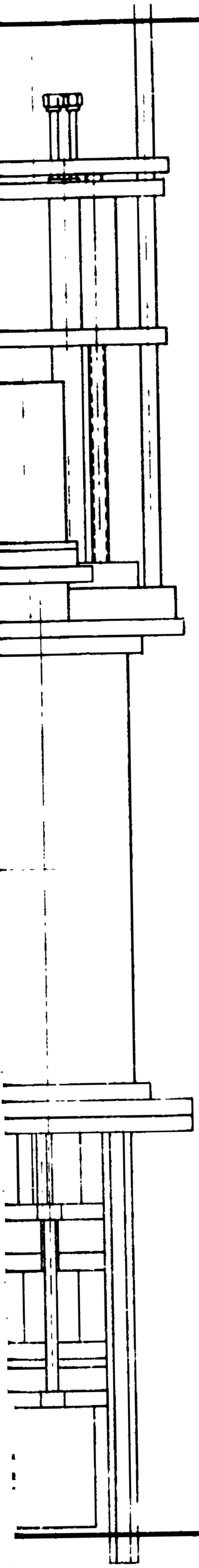


Figure 20. Assembled Thin Rod Zoner
Compcnents and size are shown in Figure 25.
Total weight is 84 pounds.


FURNACE TRANSPORT ASSEMBLY
UPPER ROTATION MOTOR
FURNACE TRANSPORT MOTOR
FURNACE ASSEMBLY
VACUUM CHAMBER
LOWER TRANSPORT MOTOR
LOWER ROTATION MOTOR
5.00 IN. 12.7 cm
14.50 IN. 36.83 cm
47.44 IN. 120.50 cm
14.00 IN. 35.56 cm
1.00 IN. 25.4 mm
2.12 IN. 5.38 cm
13.25 IN. 33.66 cm
6.25 IN 15.88 cm



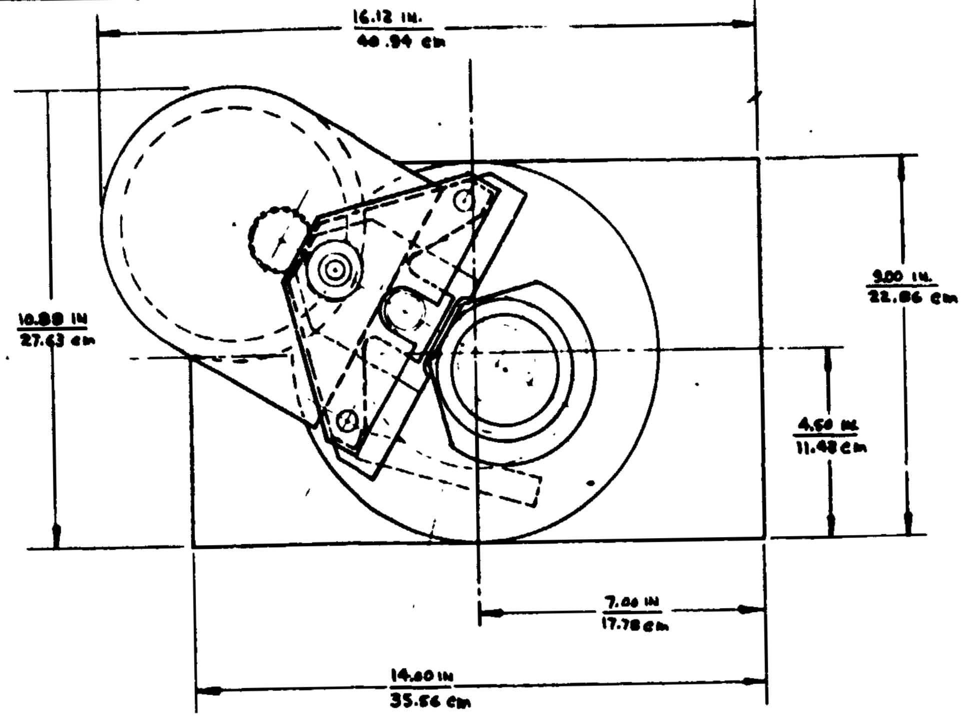


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Figure 21. Thin Rod Zoner Profile

Heights and widths can be changed by foreshortening plates and rods used for mounting in the Test Chamber and the placement of mechanical drive components.

2 FOLDOUT FRAME

INCHES

WESTECH SYSTEMS, INC.

SCALE: HALF SIZE

DATE: 7-29-85

THIN ROD ZONER

983-81001

needed for the upper transport, the upper shaft that holds the silicon feed rod does not translate. Instead, the furnace moves up and down within the vacuum chamber.

The mechanical movements are provided by four motors. The motors are seen encased in cylindrical cannisters in Figure 21. The upper rotation motor sits over the center of the furnace, with its rotary seal provided by an o-ring seal. The upper and lower shafts, which hold the chucks holding the silicon feed rod (upper chuck) and seed rod and growing crystal (lower chuck) are water cooled, using o-ring sealed cavities to get the water (coolant) into the rotating shafts. This is done to assure that the heat is carried away properly by the coolant and that the rotation seals have a long life. Growth runs will be done with and without coolant during the experimentation stage and the need for coolant thoroughly analyzed. Initial runs show promise that the cooling may not be necessary. If this is proven, the shaft seal designs can be simplified for the flight model.

The motor assembly to raise the furnace inside the vacuum chamber is at the upper left. This raises and lowers a tube, attached to the furnace, which can be seen in Figure 26 . The protective cannisters provide several functions. They are both an electrical ground and an EMF shield. They contain a motor, an optical encoder and a gear reducer. Electrical connectors are located inside, for the removal of the components, and these connectors are connected to insulating feed throughs. A protective air atmosphere, usually needed for operation of the motor's brushes, can be maintained if it is desirable to have an inert gas in the outer cavity (EAC). These cannisters are o-ring sealed.

The lower rotation and translation motor assemblies are at the bottom of the vacuum chamber.

The motors chosen are a small permanent magnet type with extremely smooth motion. These units were not available with a tachometer. To allow for easy computer data keeping, an optical digital tachometer (encoder) was selected and mounted.

Velocity control is provided by a bi-directional hybrid power integrated circuit (see Figure 22). An integrating differential amplifier provides the error summing function. Analog switches provide the switching of feedback and reference to reverse direction. Digital voltmeters provide translation velocity display as well as rotation velocity. The motor controllers are shown in Figures 23 (front panel) and 24 (chassis and components). This design is easily integrated into a microprocessor control, where the controllers and microprocessor can be miniaturized and fabricated for space environmental conditions and size and power requirements.

Figure 21 is an overall assembly drawing of the Thin Rod Zoner, showing maximum dimensions. The present weight of the unit pictured in Figure 20 is 84 pounds. It is estimated that 10-12 pounds could be easily removed by designed metal stock removal without affecting structural integrity. The chamber and cannisters are 304 stainless steel, while many other parts are aluminum.

The test chamber is shown in Figure 25. Its purpose is two-fold: 1) to facilitate easy startup under a variety of gas and/or vacuum test conditions and 2) to determine heat loads under conditions similar to the zoner being housed in an EAC. In the first case, the gas or vacuum zoning environment can be selected for microgravity experiments and then the suitable

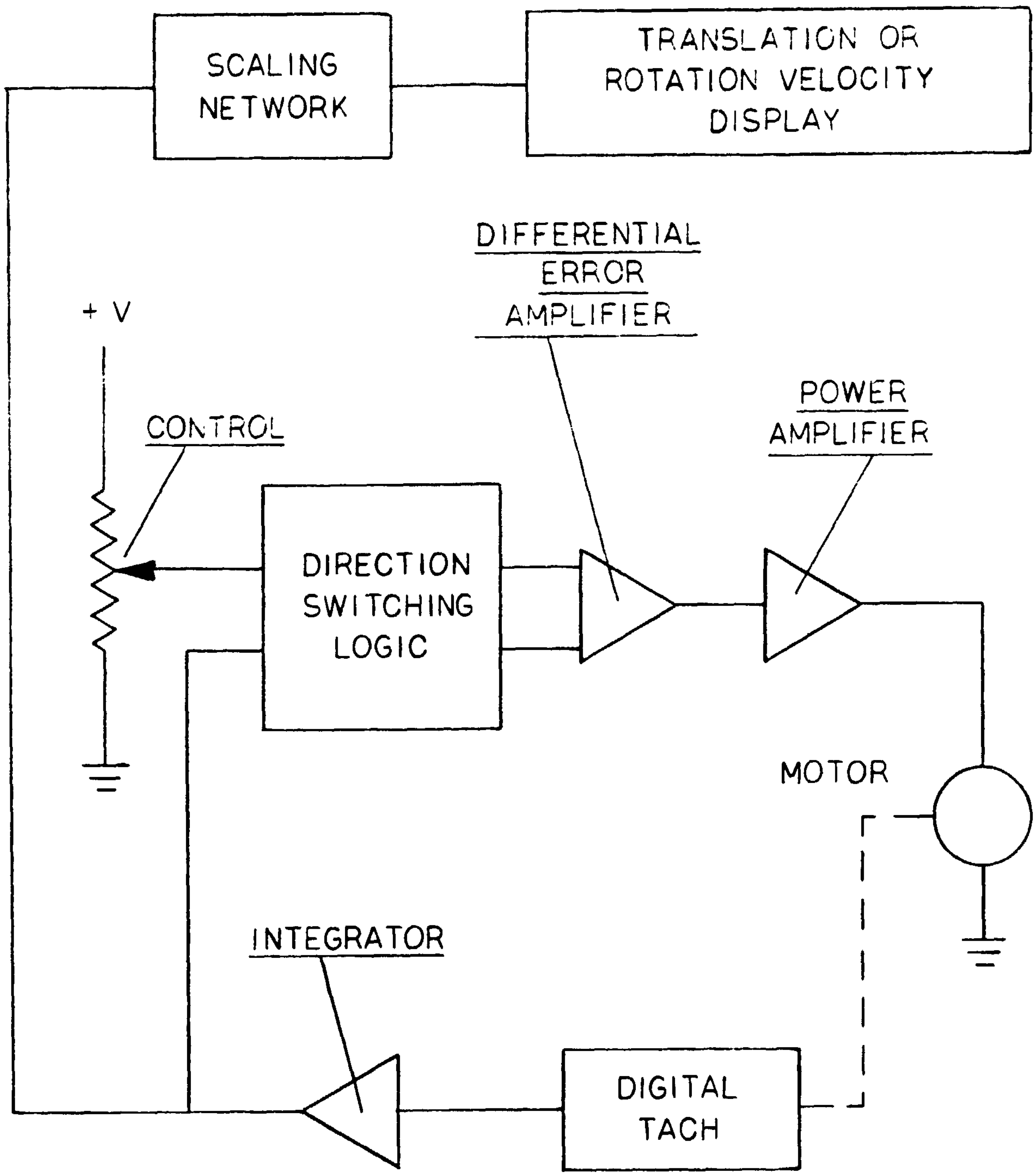

Figure 22. Block Diagram of Thin Rod Motor Control

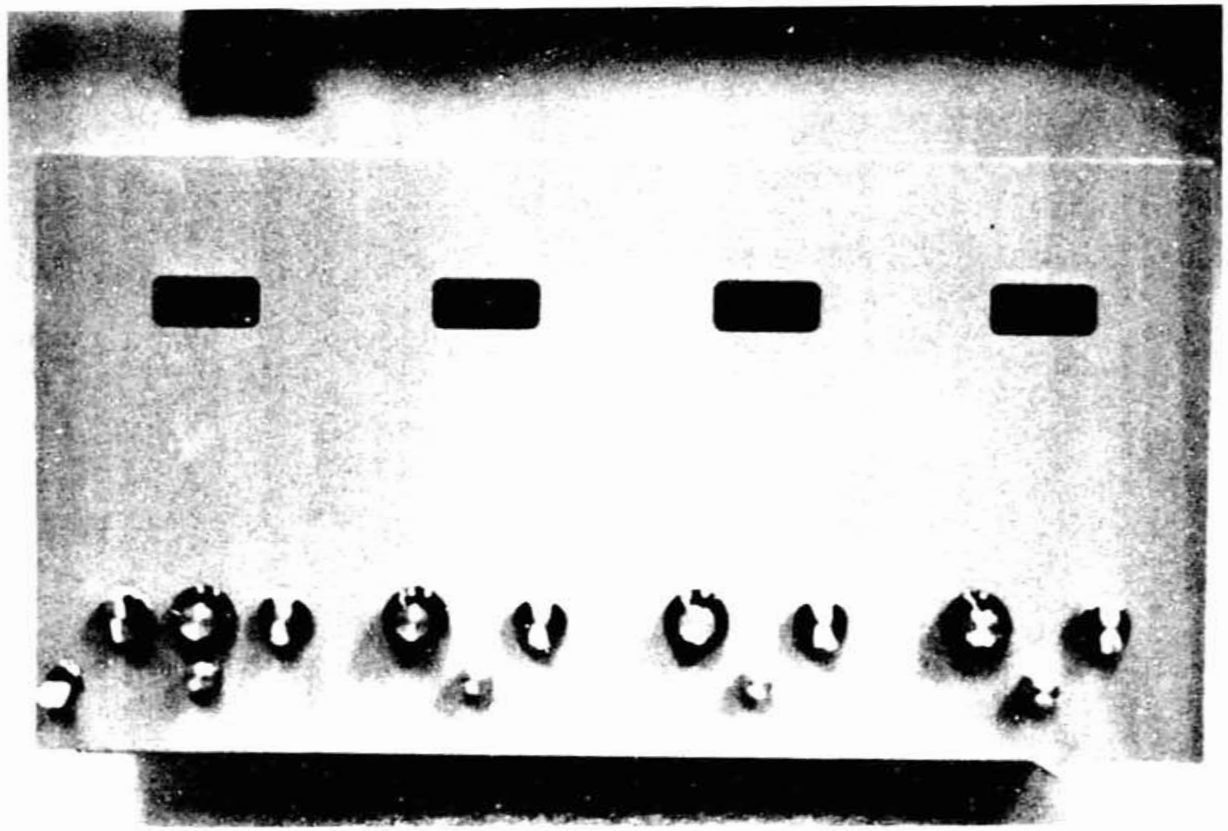

Figure 23. Motor Controller
Digital speed displays are at top and manual contols at bottom. (Labels are to be added.)

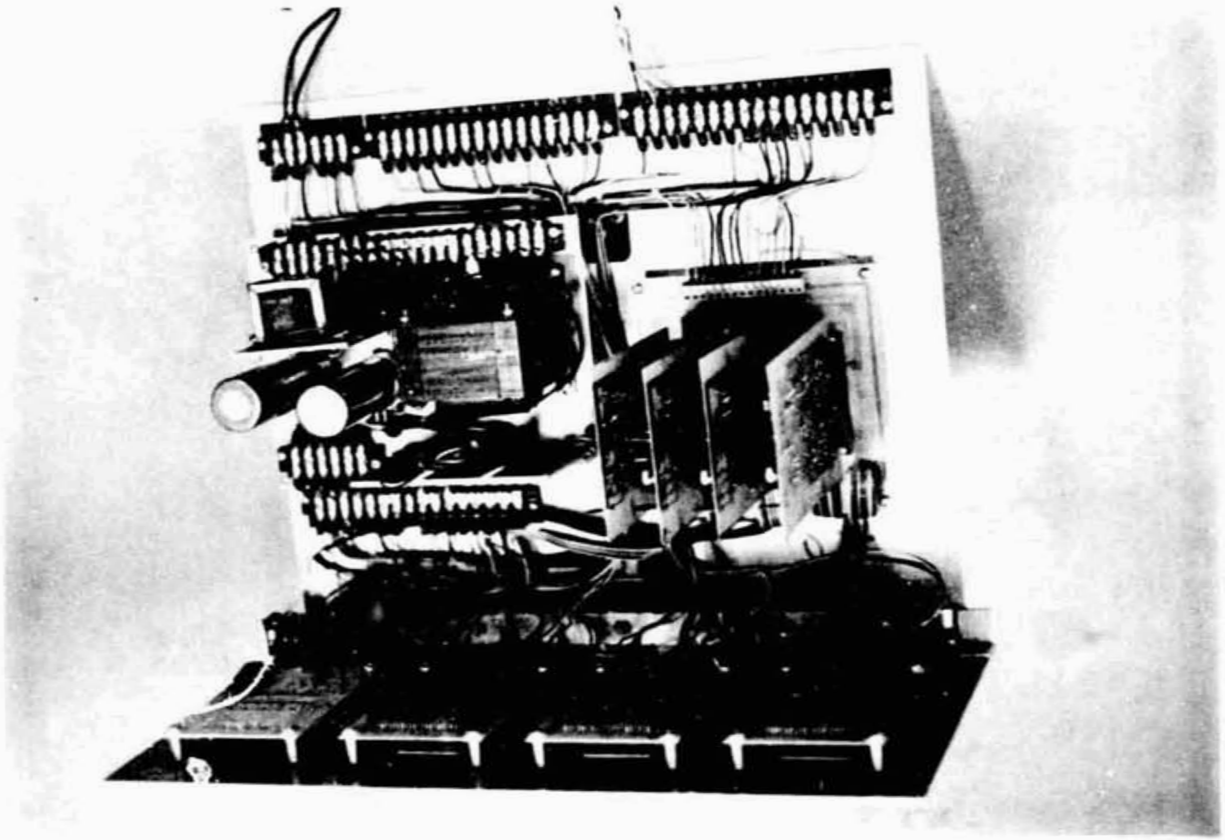

Figure 24. Motor Controller Chassis
Display P.C. cards are at bottom,
with controller circuitry on the right.

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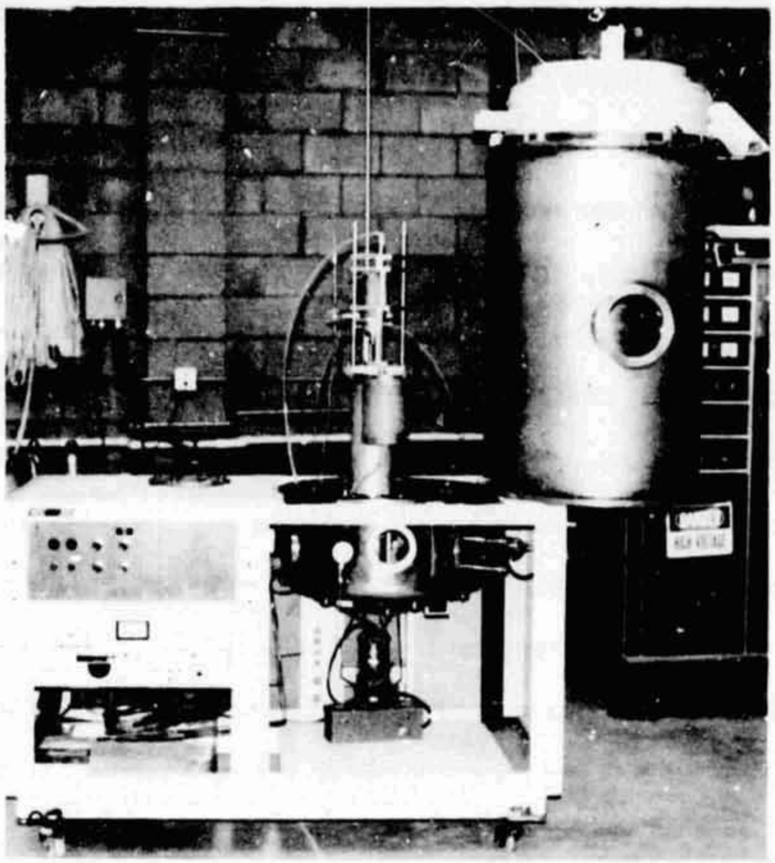

Figure 25. Thin Rod Zoner in Test Chamber

equipment can be added or developed. Wiring and coolant lines and connections are also similar to those expected in an EAC.

B. HEATER DESIGN

The concept of a resistance heated, hot wall furnace is to design the wall temperature profile to suit the method of crystal growth and the material being grown. With the ADSF, and many other Bridgman growth furnaces, a hot and flat zone is next to an insulating (adiabatic) and narrow zone, which is adjacent to a colder zone. Since the latter zone is primarily for heat extraction, cooling by a water cooled copper block is often suitable (ADSF). In float zoning, where only a narrow zone is molten, the end zones must be tailored to a lower temperature profile, with a hot and narrow center zone. Thus, a new heater profile has to be developed. While it was desirable to work with commercial heater manufacturers (such as Artcore), problems with these heaters forced the program to develop a heater design capability midway in the contract year. Close communication with MSFC led to making use of NASA's experience in this area.

The basic furnace design was patterned after the ADSF. The method of cooling the furnace ends with water cooled, copper plates, of housing in an aluminum cylinder, of using thick and low heat conductivity insulation and several other features are the same as the ADSF. The nature of the heaters and thermal profiles and the higher temperature of operation are, however, quite different for a float zoner. Thermocouples are used for reading the temperatures. The assembled furnace is shown in Figure 26. The long tube in the front is used for moving the furnace axially in the vacuum/gas chamber. It also carries all the wires and cooling lines out of the chamber. The copper end

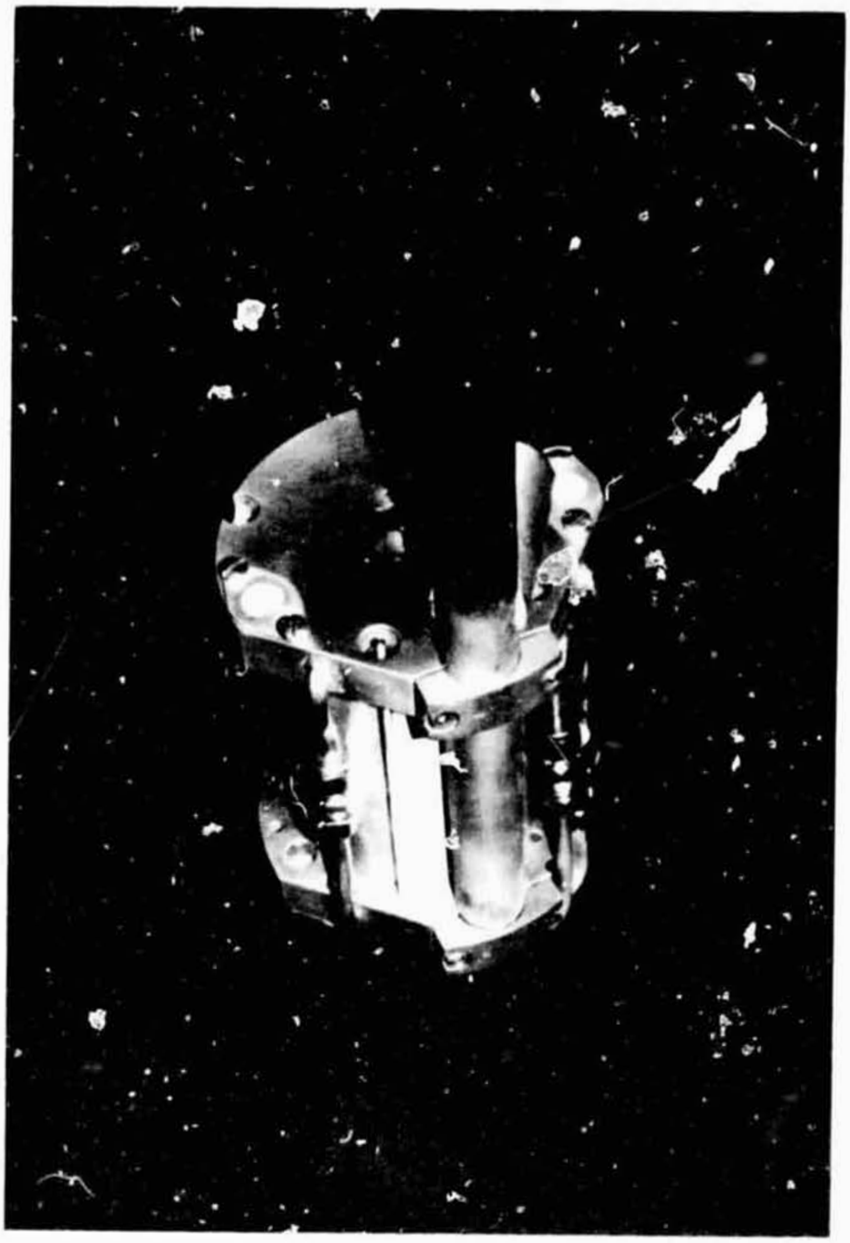

Figure 26. Heater Assembly for Thin Rod Zoner
Tube for axial motion of furnace and carrying cooling lines and wires is in the foreground. Weight of this assembly is 6 pounds.

plates have machined cooling ways and also provide for holding the bushings for the guide rods which align the furnace and for securing the wire leads (thermocouple and heater) which pass through the end plates.

The insulation chosen is a Zircar® zirconium oxide expanded foam insulation, with very low thermal conductivity. It is good to the high temperatures expected with a ZrO_2 heater. The insulation is made of four concentric, machined (by Zircar, Inc.) cylinders, separated with ZrO_2 woven cloth (see Figure 27). On each end there is a flat cylindrical plate and a sheet of woven cloth. Thermocouple and heater wire leads are brought out through holes in the end insulating plate.

The wall temperature and temperature gradient that was required for zoning silicon was unknown and needed to be determined either by modeling or emperically. The combined effects of heat flow down the silicon rods on each end of the melt, of the reflectivity of silicon to radiant heat and of gas ambient cooling was unknown. Silicon has always been zoned with r.f. heating, so there was no experience to call upon. An initial modeling calculation for germanium by Naumann and Cothran (MSFC) (presented at the February 23, 1982 Float Zone Workshop in Phoenix) indicated that the heater wall needs to be several hundred degrees hotter than the melting point.

An analytical modeling effort, similar to Naumann's model for the Bridgman furnace which had been completed, was proposed by Larry Foster of SAI. The specific goal of that effort is to define the heater wall profile which will give a flat growth interface. A flat growth interface and flat isothermal lines in the cooling crystal will minimize hoop stresses

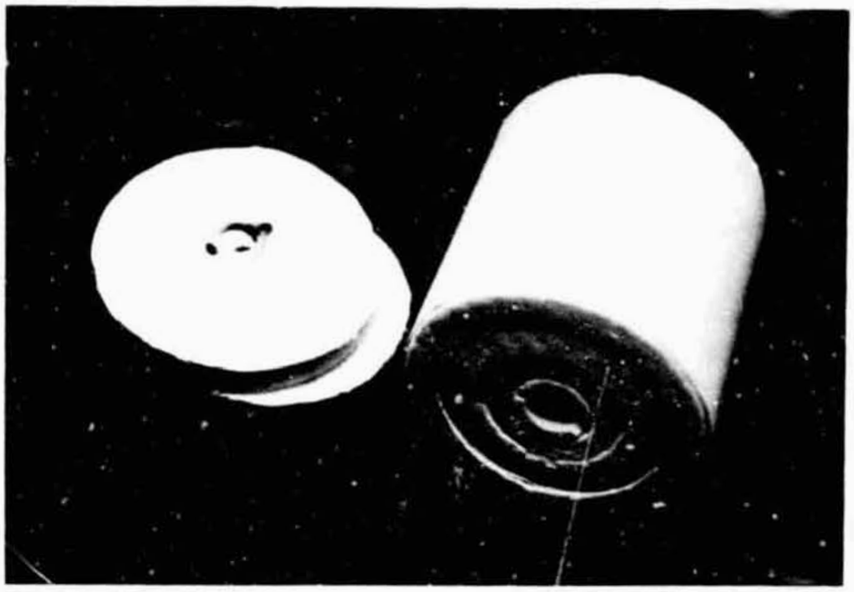

Figure 27. ZrO_2 Insulation Assembly for Furnace
Expanded ZrO_2 cylinders separated by ZrO_2 woven cloth.

Figure 28. ZrO_2 Resistance Heater (Artcore)

and, in turn, minimize the formation of bands of vacancies and subsequently formed vacancy clusters. Start of the Foster effort was delayed such that the thermal design information from it would come after the furnace was built and initially run. That information will be used to optimize the heater design and help explain the characteristics of zoning the silicon crystals and their crystallography.

Since the Naumann and Cothran modeling pointed to temperature needs much higher than the melting point, it was decided to use a heater capable of 1800-2000°C. The choices were a) a refractory metal, such as tungsten (or graphite) or b) a refractory oxide, such as zirconium oxide. A refractory metal would need to be wound on a refractory oxide muffle, since using a refractory metal muffle would not allow for steep enough gradients. It was thought that reaction with ceramic oxides or oxygen and water vapor in the gas (Argon) or vacuum atmosphere would oxidize the tungsten. Experience at MSFC with Artcore's ZrO_2 heaters led to considering them. A meeting was held at Artcore, who indicated how they could design a thermal profile into a graded-cross section heater wall. An initial heater was obtained from MSFC for furnace startup. The initial heater used in this furnace is shown in Figure 28. Nichrome preheaters were fabricated for heating the ZrO_2 to a temperature to where it will pass current and heat itself. When designing and assembling the furnace and working on startup problems, communication with Artcore was difficult. This and subsequent failures of the heater led to the conclusion that working with Artcore (which has a proprietary design and fabrication method) would not lead to a successful furnace development in the time required.

Problems with the zirconia heater led to reconsideration

of precious metal wire heaters. While the Pt heater of the ADSF was determined inadequate for going to a high enough temperature, the range of temperatures afforded by the platinum/rhodium alloys, or even rhodium or iridium, could provide temperatures as high as the zirconia heater (see Table 3). Alloys of platinum and rhodium are recommended for the 1600-1800°C range (Englehard). Preliminary design was done with chromel and, going to higher temperatures, Kanthal wire. These are easy to work with and much less expensive than Pt/Rh alloys, which must be ordered to the desired diameter.

The concept for the heater profile is to heat up the feed rod with a gradual gradient, then supply just the heat needed to provide the heat of fusion and keep the melt from solidifying over a narrow melt region, then allow the growing crystal to cool in a gradual manner. This is expected to a) minimize the superheating of the melt by keeping the ΔT in the melt as low as possible and thereby drive Marangoni flows as little as possible and b) provide for flat isotherms in the grown crystal to minimize crystallographic defects. This can best be accompl ~hed by using multiple winding levels, with each level being capab.. of separate current control. A burned out ADSF Pt heater was used as a model. Since no winding design was available, it was x-rayed and used this as an initial information point. Several designs are indicated in Table 4. One winding is designed to provide a spike at the desired hottest area, like a tickler coil. Figure 29 shows one of the Kanthal test heaters, wound on an Al_2O_3 muffle and insulated with zirconia cement. The latter was used for its high temperature capabilities, at t! recommendation of Zircar,

Table 3 Precious Metal Heater Materials

Property \ Metal	Pt	Pt 10% Rh	Pt 20% Rh	Pt 40% Rh	Rh	Ir
Melting Point °C	1769	1830	1860	1920	1966	2410
Resistivity* at						
20°C	65	115	125	105	30	
1000°C	250	280	260	240	65	
1300°C	295	325	300	290	-	
1400°C	305	335	310	305	-	
1500°C	320	350	320	320	-	
1600°C	350	370	-	335	310	

* ohms/circular mil ft.

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Figure 29. Kanthal Wire Test Heater
Two windings, connected in series, on a Al_2O_3 muffle with ZrO_2 insulating cement.

Table 4 Resistance Heater Designs

No.	No Winding Layers	Flat Hot Zone (inch)	Layer No.	Winding Configuration # Turns				Total No. Turns	Comments
				Left	Center (hot)	Center Right	Right		
1.	2	1/2	#1	1	3	3	1	7½	
			#2	- - - -	- - -uniform- - -		- - - - -	15½	
2.	3	3/8	#1	1	2	-	1	4	
			#2	5	3	-	3	11	
			#3	- - - -	- - -uniform- - -		- - - - -	9½	
3.	2	3/16	#1	11	2	-	14½	27½	
			#2	5	2	-	5½	12½	
4.	2	5/16	#1	8½	3	-	12	23½	Pt 10% Rh profiled in Figure 30
			#2	4	1½	-	5½	11	
5.	2	3/16	#1	9½	2	-	12	23½	Heater now being fabricated.
			#2	3	2		5	10	

Al_2O_3 muffle is 4 inches long. For designs 1 through 4, the I.D. is 0.375 inches and O.D. 0.50 inches. For design 5, the I.D. is 0.50 inches and O.D. 0.75 inches.

Inc.

A Pt-10% Rh heater, using 0.032 inch diameter wire was constructed and profiled. Initial bench profiling was done by insulating the heater with alumina wool. Axial profiling and working with thermocouples is easiest in this configuration. Profiling in the furnace assembly provides the profile seen in Figure 30. The windings were initially designed for a 3/16 inch flat, hot zone, similar to that of a Kanthal heater seen in Figure 29, with the hottest zone displaced 1/2 inch below the center of the 4 inch long windings. X-rays of the heater windings (for the heater profiled in Figure 30) show a 5/16 (or 0.31) inch length of maximum windings centered 1/2 inch below the center of the heater. Two wire layers are connected in series. The profile in Figure 30 shows the maximum temperature is 0.46 inches wide and located ¼ inch from the heater source. This shift is thought to be due to an influence which tends to center the maximum temperature in the center of the windings. The next heater has been designed to concentrate the windings in a narrower zone (3/16 inch), closer to the center (13/32 or about 3/8 inch) and independently powering the two layers.

The heater controls were originally designed similar to the MSFC controls, using a Research Incorp. Phaser and a proportional controller. With the temperature-resistance characteristics of Pt-10% Rh, a non-phase-pulsed method was preferred and a variac control has been used with a.c. power.

The initial thermocouples were W-Re. We could foresee problems with slow reaction with the cements (since we have the thermocouples in contact with the heater) and gases in the

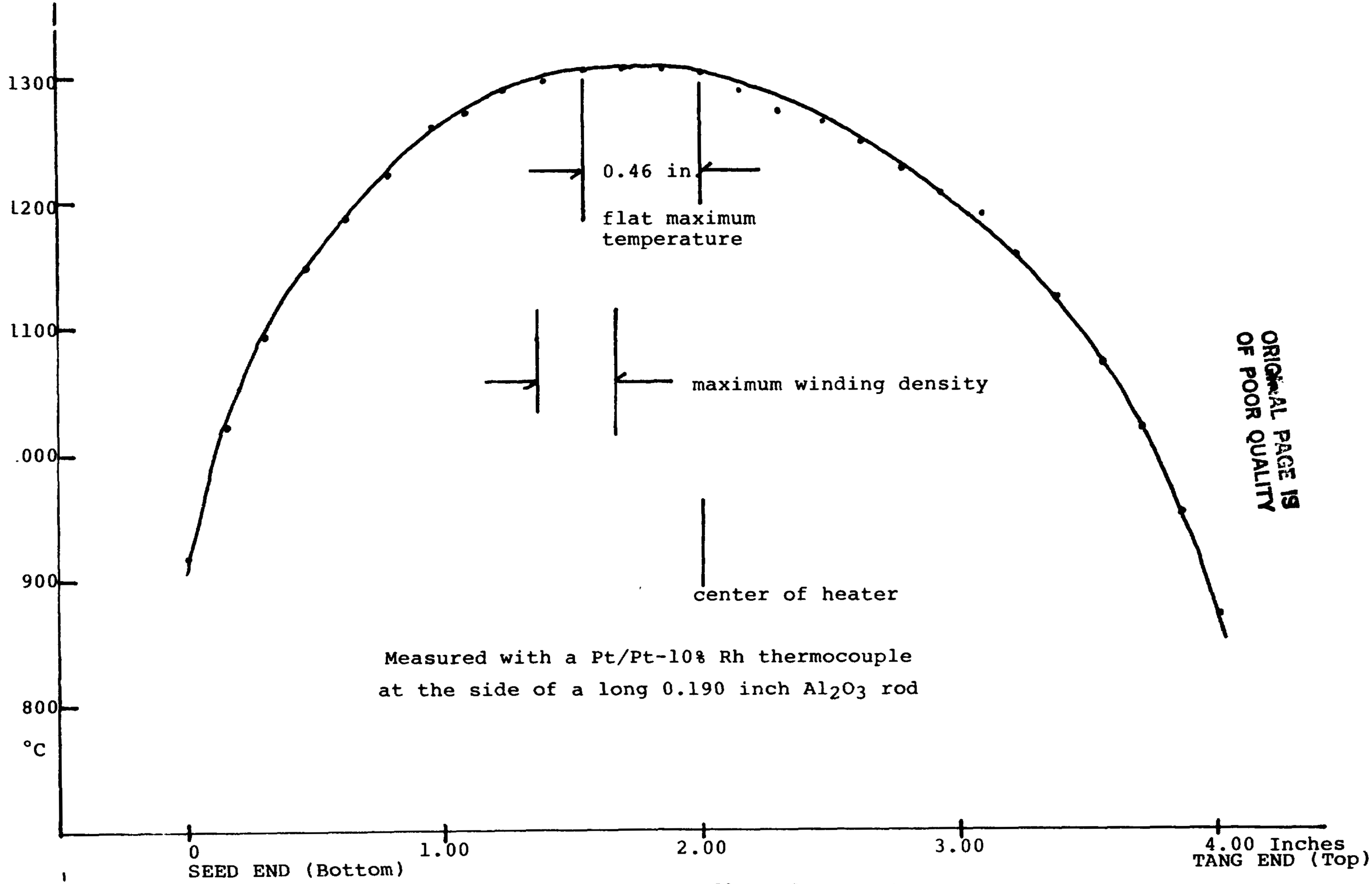

Figure 30. Thermal Profile of Pt Heater
Maximum temperature during profiling was 1310°C

atmosphere. The switch was made to Pt-Pt/10% Rh at 5 mil diameter. Future thermocouples will be made with Pt-Pt/13% Rh at 20 mil diameter for more stability. They will be cemented directly to the outside diameter of the Al_2O_3 tube (between heater windings) to insure intimate contact with the tube. Several thermocouples will be spaced radially around the hot zone (for added protection) and two at each end of the heater.

The heater profiling (as shown in Figures 30 and 31) is measured with a Pt-Pt/10% Rh thermocouple which is at the surface, and midway down a long Al_2O_3 tube (probe) of roughly the same diameter as the silicon to be zoned. This approximates a long silicon rod, except that the thermal conductivity is much lower for the Al_2O_3.

C. ZONER STARTUP

Startup of a new furnace design is expected to result in some initial failures and at least minor design changes, especially for untried subsystems, such as the heater. The startup of mechanical, electrical and vacuum parts went smoothly. Most of the problems were with the heater, as described below. Initial melting results with silicon are given in the next section.

The first heater failures were with the zirconia heater. After initial profiling and making sure the ZrO_2 tube heated uniformly (which is often a problem), the temperature was raised to 1300°C. The heater failed due to the cemented Pt current lead melting. A new Pt wire, which was of larger diameter, was attached to the heater cylinder with the cement recommended. This time, the cement and/or Pt reacted with the ZrO_2 and the

heater broke at 1530°C. This and previous communication problems with Artcore led to abandoning the zirconia heater.

Correlation of thermocouples and profiling has led to questions of how close the thermocouples read (the outer wall of the Al_2O_3 muffle at its center vs the profiling probe) and solutions are proposed for future work. The thermocouple at the heater center was mechanically held in a recess that was drilled into the cement. While correlation between this thermocouple and the profiling thermocouple in the heater cavity was measured within 5°C, there is always a question as to whether the thermocouple is in intimate contact when operating the furnace. Thus, future thermocouples will be cemented into the heater. The zirconia cement has reacted with the thermocouple wire, as evidenced from a tarnished color. Communication with Motorola personnel(7) uncovered their past experience with platinum reacting with the free silica in ceramics. The zirconia cement used here analyzed as having 3.6% free silica. In the future, pure Al_2O_3 cement, with an analysis of no trace silica, will be used.

After considerable profiling and several silicon melts, the Pt/Rh heater element failed. The temperature was close to the melting point of silicon, 400°C below the melting point of Pt/10% Rh. The heater was broken apart, revealing a melting of the wire and what appeared to be extensive interaction at the location where the heater was electrically open. This is thought to be an interaction with the free silica. A silica free cement (and alumina muffle) and Pt-40% Rh (with 90°C higher melting point) will be used for the next heater design.

One silicon melt fused to the muffle wall. Analysis showed that the water cooled upper chuck was slightly misaligned, apparently happening when a leak in the chuck was repaired. The chuck was remachined and an exact alignment check routinely introduced. The design of these water cooled chucks and their seals will be reviewed by looking at the heat carried away (or temperature rise when no coolant is used) when the desired atmospheric conditions have been established (i.e., the conditions to be used for microgravity experiments).

The need for thermal expansion clearances and the intolerances in ceramic parts leads to an alignment of parts which needs improvement. The earlier recommendation (by Coors and Artcore) of not having a single ceramic tube going from cold end to cold end will be reviewed. Unless that now looks feasible, the ceramic tubes will be machined to be precisely self aligning, yet allow for expansion.

A thin clear quartz sleeve will be used inside a slightly larger diameter Al_2O_3 muffle. Thus, when silicon melts contact the wall, a readily replaced quartz tube will protect the heater assembly. This also has the possibility of directing an inert gas flow (as an atmospheric condition), of keeping in volatile components and of keeping out contaminants that might come from the furnace. Discoloration of a short length of the silicon rods indicates we have some outgassing from heating the ceramic or gaseous contaminants that are not yet sufficiently purged. Keeping in volatile components could lead to zoning compound semiconductors, such as GaAs or CdTe. Consideration, in the future, will also be given to using a clear, thin sapphire tube

in place of the quartz. These are commercially available, being made by the edge-film-defined method.

D. INITIAL MELTING RESULTS

Silicon was initially melted in a graphite boat by the Pt-10% Rh heater in a bench test, proving that a temperature somewhat lower than the 1620°C maximum measured heater temperature would melt silicon in a boat.

The next melting experiments were in the fully assembled furnace. A run was made in a vacuum of 10^{-4} torr. The superior insulation of the furnace led to melting at a much lower current than was needed to obtain melting on the bench (< 10 amps vs. 15.25 amps for 1630°). Less than 240 watts (at 10 amps) melted a zone length of 33.4 ml (98 mm^3). The silicon melt froze to the Al_2O_3 muffle wall and had to be carefully ground out with a small diamond cylindrical grinding tool.

The furnace was further profiled and the end of a silicon rod carefully melted (at 2.5 psi Argon). Melting started when the center thermocouple (in contact with the outside of the Al_2O_3 muffle) indicated 1430°C. At between 1435 and 1440°C, a 1.5mm high portion was melted (4.7mm^3 or 10^{-2} gms.) This indicates melting of the silicon when the temperature is only 10-15°C higher than the silicon melting point (depending on the accuracy of the thermocouples - and there is no convenient way to calibrate them at this temperature). It also indicates that the silicon rod is not extracting heat faster than the hot wall heater is supplying it to the solid silicon rod.

The power to melt 5mm diameter silicon in 1 atm. Argon

appeared to be about 170 watts.

The use of moderate vacuum (between 10^{-2} torr and 10 torr) would seem to be ideal. The vapor pressure of silicon and Pt is each around 10^{-3} torr at 1420°C. Thus, 10^{-2} torr would avoid vaporization and deposits.

The next run was made at 0.1 torr. At 88 watts (87.76 watts at 10.97 v. and 8.0 amps), an 81mm length of silicon was melted, which then solidified on the muffle, breaking it. At the same time, the heater failed (opened), because of the reaction to silica mentioned in the last section.

The power to produce a given temperature at a given current is shown in Figure 31 for a current of 11) amps for the Pt-10% Rh heater. 164 watts gives heater temperature of 1420°C. If 1430°C is needed to melt during zoning, it would require 165 w.

In conclusion, while a successful zone pass has not yet been made with silicon, the power to melt silicon has been determined to be less than 200 watts at a voltage at or below 15 v.a.c. in 1 atom Argon and < 100 watts at a voltage of < 11 v.a.c. at 0.1 torr vacuum. This is certainly within the voltage and power budgets of a Hitchhiker carrier on the shuttle. The next heater, along with the procedures described herein, is expected to result in a stable and controlled zone configuration.

E. PROGRAM OF INITIAL ZONING PARAMETERS

Zoning in a Thin Rod Zoner must be controlled by a different method than an r.f. zoner, where the operator can observe and even measure what the growing crystal is doing. In the Thin Rod Zoner, it is impossible to observe the crystal or melt; thus

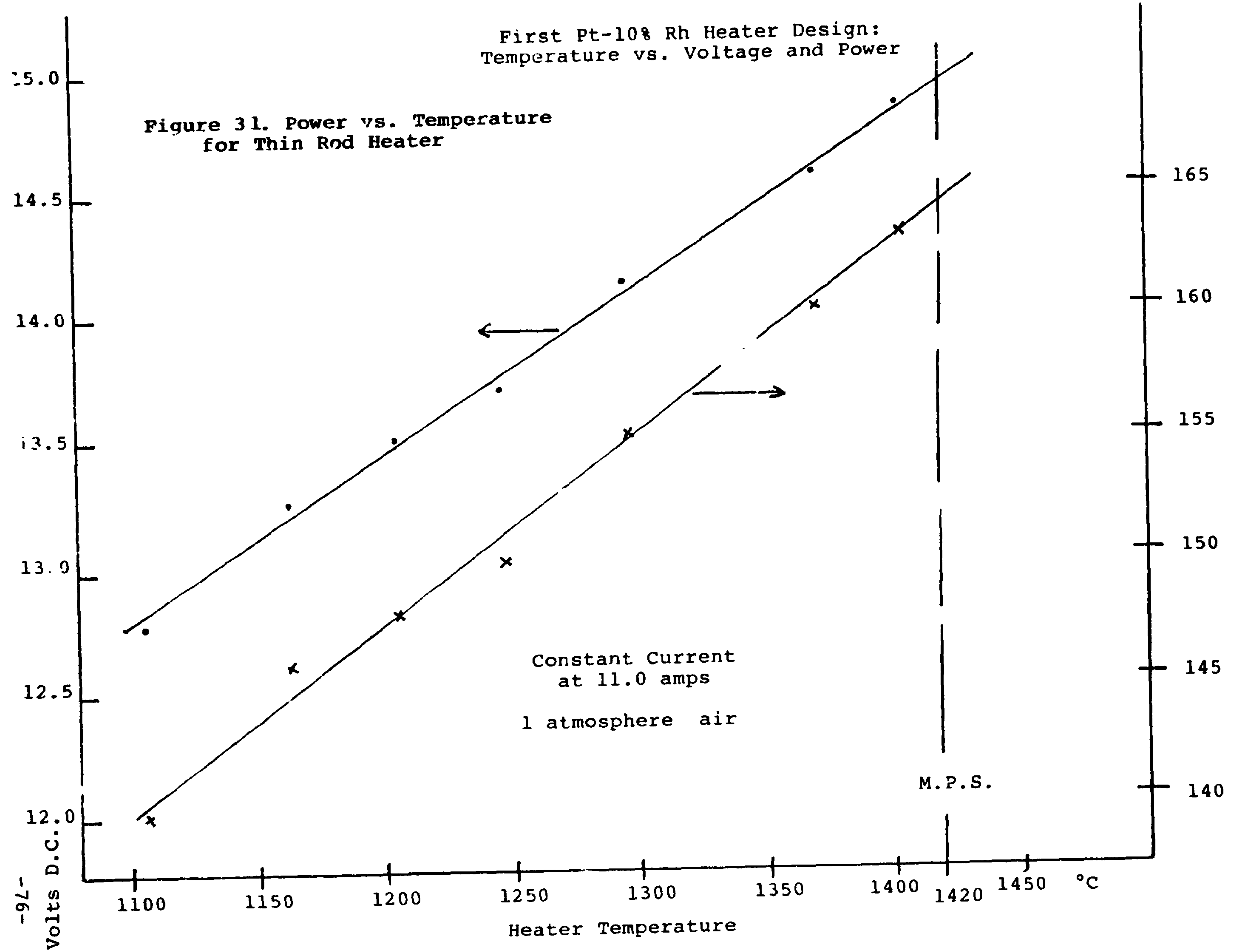

Figure 31. Power vs. Temperature for Thin Rod Heater

the growth will have to be done by programming speeds and power levels. This is straight forward when zoning at constant diameter. The program must be developed for starting the growth from the seed, necking down for dislocation-free growth and making the necessary transition shape going out to full diameter.

Table 5 shows the times and axial speeds used at one given power setting to go from the seed touching the melt to coming out to a full 1.0 cm diameter. The point of reaching 5mm diameter (for initial flight experiments) is also shown. The speeds were recorded on a chart recorder, while timing with a watch and writing down observations.

The approach to programming the initial stages of crystal on the Thin Rod Zoner will be to use the results of this run, plus several more. The speed excursions will be smoothed and the speeds averaged. This will then be tried on the r.f. zoner, in place of the operator's instinct and normal control reaction. After optimizing this procedure, the program will then be put into the Thin Rod Zoner controls and tried. Since the conditions are much different in the Thin Rod Zoner, it is expected that this program will have to be modified substantially before the right conditions are found for good crystal growth.

This growth speed of 5mm/minute is a relatively fast zoning speed, which resulted in dislocation-free crystal growth, leads to a short time needed for growth. With a 1 cm diameter transition (time to reach full diameter) taking 4 minutes, a 2 cm. long crystal at 1 cm diameter could be grown in 8 minutes, a time duration which might be possible in a sounding rocket. (Most SPAR flights now give 6 minutes of microgravity).

Table 5 Thin Rod Zoning Parameter Program

Times and Speeds at $\geq$ 5mm/min zoning speed

TIME		SPEEDS		EVENT
Min	Sec	Upper mm/min	Lower mm/min	
0	00	0	0	Starting to melt top rod
0	21	0	0	Put seed into melt
0	25	0	0	Seed starting to couple (heating up)
0	28	0	0	Seed fully coupled
0	40	3.5	13.5	Starting to neck for dislocation free
1	00	3.5	20	Growing neck
1	30	3.5	23	Growing neck
2	03	1.8	10.0	Increasing diameter
2	12	var *	4.8	
2	21	4.7	var	
2	30	var	8.5	
2	33	11.0	8.5	
2	43	9.3	8.5	
2	48	9.7	8.5	at 5mm diameter
3	30	7.3	5.8	
3	54	7.7	5.8	at 10mm diameter dislocation free

* variable -- operator is changing up and down

rotational rates: top rod 0
bottom seed 15 rpm

IV. RECOMMENDATIONS

The benefits of float zone growth of crystals in microgravity can be projected to rely on these areas of concern:

a) thorough knowledge of how a crystal grows on earth and its subsequent properties

b) the role Marangoni flow will play on fluid flows; predict the result of these flows and propose methods to reduce or eliminate the flows

c) from these, projecting the changes (and benefits) by going to microgravity

d) developing a growth system which can carry out the microgravity experiments within the available power, cooling, size and weight capabilities of early systems (such as the Hitchhiker carrier on the Shuttle).

Silicon has been selected as the high temperature model which is representative of commercially important semiconductor crystals. Each of the above areas will be discussed with respect to what has been accomplished and where we are today. Recommendations will be made to develop each area further toward reaching the goal of characterizing the feasibility and advantages of float zoning in microgravity.

a) A detailed understanding of how float zoned silicon grows, including all of the heat and fluid flows and their characteristics, is beginning to emerge. Since this involves new modeling efforts and new measuring techniques, which require time to develop, the progress is not rapid. One area where

modeling is still to be completed is on transient flows. Transient behavior, as characterized by striations, has been studied experimentally in the previous contract and this contract. The transient temperature excursions required new measuring methods and work has been done on these on this contract and initial measurements presented. These now need to be studied in the flight experiment diameters (5mm) and heating method. Thermal profiles in the solid are presented.

b) The role of Marangoni (surface tension driven) flow and comparison to buoyancy melt flow needs to be determined. The Marangoni coefficient is still in the process of being measured (by Hardy of N.B.S.). Indications are that significant Marangoni flow is expected in microgravity for any reasonable melt temperature gradient. Whether this flow is laminar or oscillatory is not known. Attempts have been made to observe this flow in thin slices, but more controlled experimental work is needed. If flow can be experimentally determined, then the arresting of that flow with surface layers can be attempted and the results observed.

c) Understanding the growth dynamics and the role and/or control of Marangoni flow should allow prediction of what improvements can be made in (silicon) crystals prepared in microgravity. Even if the improvement in silicon were to not be significant enough to warrant the increased costs of producing in space, several significant benefits could occur. The use of more uniform or crystallographically better crystals could be used as calibration samples for the commercial semiconductor

industry and for exploring new generations of devices. The role of gravity, Marangoni flows and transients and their control is also a major process concern in commercially growing silicon on earth. These predictions can only be made when a) and b) are understood. One of the best ways of predicting these, and the real proof, is to do meaningful growth experiments in microgravity, thus requiring an experimental growth zoner (item d)).

d) An early experimental growth system must fit within the available resources of the space carrier systems, such as a MEA or Hitchhiker carrier on the Shuttle. A resistance heated zoning unit for small (5-7mm) diameter silicon growth has been designed, fabricated and put into operation. The utility requirements have been shown to be within those provided by MEA or Hitchhiker. The zoner has been designed to fit the space and weight limits of an EAC and has been designed to interface to control systems which can be readily developed for flight. Further work must be done on optimizing the heater design, developing the zoning program and gathering zoning data. The equipment should be developed with all the functional controls and auxiliary equipment to have it operate automatically, as it needs to for flight experiments and to gather data on ground based growth as if it were doing the experiments in microgravity.

It is recommended that the ground based research in areas a), b) and d) be completed with an additional year's efforts. This will provide the exact microgravity experimental conditions

and the equipment design needed to fabricate the flight hardware and prepare for the flight experiments.

Specific major portions of the next year's efforts are:

1) Build a small, radiant heated slice zoning apparatus and study Marangoni flows and their control by various surface changes.

2) Optimize the Thin Rod Zoner heater design for control of zoning conditions. Determine the recommended zoning parameters and measure power, heat flows, etc. Measure the crystal properties of crystals grown under various conditions.

3) Further develop the sample holders, including the need to cool or not, the ability to pass a peltier pulse, the ability to withstand environmental conditions on the Shuttle and compatibility with sample changing.

4) Control the Thin Rod Zoner (heater and motor controls, gas environment, etc.) with a microprocessor (off-the-shelf), programming in the zoning parameters.

5) Develop the hardware concepts and demonstrate the feasibility of changing samples within a single shuttle flight. Characterizing the advantages of microgravity processing requires the analysis of silicon grown under a wide variety of axial and rotational speeds. For undoped silicon, this will require several ingots, each with several parameter changes. Relating to practical commercial or device needs requires doping, which will require more ingots. Without a sample changer, only one ingot could be done per flight and one or two flights

per year.

6) Key components of the furnace and the samples should be tested for vibration and shock conditions of the shuttle (especially during take off) before the furnace design is finalized. The natural frequency of the furnace should be ≥ 35 H_z (Hitchhiker specification). The silicon rods and ceramic furnace parts should be either not affected or properly supported so that breakage will not occur.

7) Study thermal profiling and studying melt thermal transients at the 5mm diameter proposed for the initial flight experiment. Study the growth interface shape (using peltier pulsing) as a function of growth speed and rotation rate at 5mm diameter.

List of References

1. KERN, E.L. and GILL, G.L., "Final Report Contract NAS8-34542" June 17, 1982.

2. D. SCHWABE, F. PREISSER & A. SCHARMANN, Paper lAF-81-149, XXXII Congress of International Astronautical Federation, Rome, Italy (Sept. 1981).

3. C. CHUN and W. WUEST, Paper IAF-81-128 XXXII Congress of International Astronautical Federation, Rome, Italy (Sept. 1981).

4. J. VERHOEVEN, p. 147 NASA Conference Publication 2226 "Float Zone Workshop", Sept. 1981. Ed: E.L. Kern and E.K. Cothran.

5. R.A. LOGAN and W.L. BOND, J. Appl. Phys. 30 3 p. 322 (Mar. 1959).

6. T. SUREK and B. CHALMERS, J. Cryst. Growth 29 (1975).

7. BUD KRIEISEL, Semiconductor Sector, Motorola, Inc. Phoenix, Ariz., private communication.

8. CdTe growth efforts at Hughes Aircraft under the direction of E.L. Kern. See U.S. Army reports K-80-C-0004 by S. Sen and C.J. Fischer.

9. M.W. GEIS, H.I. SMITH, B. TSAUR, J.C.C. FAN, D.J. SILVERSMITH and R.W. MOUNTAIN, J. Electrochemical Soc. 129 12 p. 2812 (Dec. 1982).

10. H.W. LAM, R.F. PINIZZOTTO and A.F. TUSCH, JR., J. Electrochemical Soc. 128 9 p. 1981-86 (Sept. 1981).

11. R. EMEIS, Z. NATURFORSCH. 9a 67 (1954).

12. "Zone Melting", W.G. PFANN, Robert E. Krieger Publishing Co., Huntington, N.Y. (1978) and W.G. PFANN, Metallurgical Reviews 2 29 (1957).

13. "Floating Zone Silicon", W. KELLER and A. MUHLBAUER (Marcel Dekker, Inc., New York (1981)).

14. R.A. BROWN, presentation material, Float Zone Working Group Meeting, MSFC, Huntsville, Ala. March 23, 1983.

15. R.W. JOHNSON, J. Appl. Phys. 34 352 (1963).